N-63-2-5

Development and Production of

IMPROVED MOLYBDENUM SHEET

by

POWDER METALLURGY TECHNIQUES

Prepared Under

U. S. NAVY,
BUREAU OF NAVAL WEAPONS

Contract NOas 60-6018-c

850150

FINAL REPORT

1 October 1959 — 30 September 1962

SYLVANIA ELECTRIC PRODUCTS INC.
Chemical & Metallurgical Division
Towanda, Pennsylvania

Development and Production of

IMPROVED MOLYBDENUM SHEET

By

POWDER METALLURGY TECHNIQUES

January 31, 1963

Prepared Under
Bureau of Naval Weapons Contract NOas 60-6018-c

Final Report
1 October 1959-30 September 1962

By Sylvania Electric Products Inc.
Chemical and Metallurgical Division
Towanda, Pennsylvania

Roger B. Bargainnier
Richard F. Cheney
Lauri D. Tiala

Approved By: _____
Engineering Manager
Metallurgy

_____
Chief Engineer

ABSTRACT

A powder-metallurgical molybdenum alloy was developed which, in wrought form, has properties as good as or better than those of arc-cast Mo-0.5Ti. The alloy, Mo-0.5Ti-0.03C (MTC), was selected after screening 15 molybdenum-alloy systems.

Process specifications for 40-mil MTC sheet were developed. In the as-rolled condition the sheet has an ultimate tensile strength at 1200 C of about 60 ksi. It is approximately 50% recrystallized after one hour at 1210 C, and its ductile-brittle transition temperature is below room temperature.

MTC seems to be responsive to strain-induced precipitation strengthening in a manner similar to that ascribed by other investigators to certain arc-cast molybdenum alloys. Sintering at $\geq$1850 C is necessary to develop the best properties in MTC sheet.

A process was also developed for rolling molybdenum powder directly to sheet. This work was summarized in a report issued March 31, 1961, and is not discussed further.

TABLE OF CONTENTS

TABLE OF CONTENTS (Cont.)

LIST OF TABLES

LIST OF TABLES (Cont.)

LIST OF ILLUSTRATIONS

LIST OF ILLUSTRATIONS (Cont.)

1.0 INTRODUCTION

When contract NOas-60-6018-c for the development of improved powder-metallurgical molybdenum sheet commenced, arc-cast molybdenum alloys for high-temperature load-bearing applications were already in an advanced state of development. Arc-cast molybdenum alloys with ultimate tensile strengths above 50,000 psi at 1200 C existed. No powder-metallurgical molybdenum alloy of comparable density came close to achieving that strength.

The powder-metallurgical approach to high-temperature molybdenum alloys has several inherent advantages. It is shorter and therefore less expensive than that of arc-casting. By the former process the volatilization of alloy constituents is less. Therefore, it was envisioned that the powder-metallurgical approach could result in substantial gains in the technology of molybdenum and its alloys.

The original objectives of this contract were:

a. To investigate and develop alloy compositions and processing conditions for the production of molybdenum-alloy sheet with improved properties and quality.

b. To determine the feasibility of rolling molybdenum sheet directly from powder.

c. To produce 8000 pounds of the alloy sheet in various sizes.

The degree of improvement to be obtained in the properties and quality of the alloyed sheet was not specified. However, during the contract the Material Advisory Board's Refractory Metal Sheet Rolling Panel recommended target properties for fabricable molybdenum-alloy sheet (see Appendix I). These recommended properties then became the inferred goal of Sylvania's program. It soon became evident that the development of a powder-metallurgical alloy with properties approaching these targets would involve a major developmental effort. The objectives of our program were subsequently modified by the Bureau of Weapons to exclude the production of all but a sample quantity of sheet.

Work under contract NOas 60-6018-c was conducted from October, 1959, through September, 1962. Seventeen interim reports were issued. The contents of those reports are outlined in Table I as a guide to the details of the entire investigation.

This final report summarizes our work leading to the improved powder-metallurgical molybdenum-alloy sheet, Mo-0.5Ti-0.03C. Our work on the rolling of molybdenum powder directly to sheet appears in a prior summary report.

TABLE I

SUMMARY OF INTERIM REPORTS

Contents	Report Number														
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17
Evaluation of Powders	X	X		X		X	X								X		
Rolling and Sintering Investigations		X	X	X			X	X	X	X			X	X	X	X	
Rolling Schedule Investigations		X	X					X						X	X	X	
Preparation of Alloy Sheet																	
a. Oxide Additives		X		X			X										
b. Elemental "				X				X		X							
c. Others				X				X	X	X					X	X	
d. Mo-Ti-C System				X				X	X	X			X	X	X	X	X
Evaluation of Unalloyed Mo Sheet		X	X	X	X	X	X										
Evaluation of Mo-0.5Ti Arc-Cast Sheet					X	X		X									
Evaluation of Powder-Metallurgical Alloys																	
a. Oxide Additives		X	X		X	X	X		X								
b. Elemental "					X	X	X		X		X						
c. Others					X	X	X	X	X								X
d. Mo-Ti-C System					X		X		X		X			X		X	X
Strengthening Mechanism in Mo-Ti-C												X	X	X			
Rolling Mo Sheet From Powder*					X	X	X	X	X								

* A final report entitled "Feasibility Study on Powder Rolling of Molybdenum Sheet" was issued on March 31, 1961.

2.0 EVALUATION OF BASE MATERIALS

To measure our progress in the development of a suitable powder-metallurgical molybdenum alloy, we initially determined pertinent properties on commercially available materials using our testing facilities and methods. We selected 40-mil unalloyed molybdenum sheet produced by powder-metallurgical techniques at Sylvania, and 40-mil arc-cast Mo-0.5Ti sheet produced by four suppliers. We evaluated the sheets for composition, structure, recrystallization temperature, tensile properties, and ductile-brittle transition temperature. Our testing procedures, outlined in Appendix II, generally followed those in "Recommended Procedures for the Testing of Refractory Metal Sheet Material", issued by the Subpanel on Standardization of Test Methods, MAB Refractory Metals Sheet Rolling Panel.

Our evaluation of the two base materials appears in Table II with corresponding values from the literature for arc-cast Mo-0.5Ti sheet. Our carbon, oxygen, nitrogen, and hydrogen analyses of the arc-cast Mo-0.5Ti sheets agree well with those reported in the literature. Our values for the recrystallization temperatures and 1200 C tensile properties are somewhat lower than those reported in the literature, whereas our room-temperature strengths are somewhat higher.

TABLE II

PROPERTIES OF 40-MIL BASE SHEET

Property	Powder-Met Molybdenum[a] Sylvania Values[b]	Arc-cast Mo-0.5Ti Sylvania Values[b][c]	Arc-cast Mo-0.5Ti Literature Values
Composition, ppm			
Carbon	21(23)	280(4)	100-400(g)
Oxygen	32(23)	28(4)	25(g)
Nitrogen	38(23)	14(4)	20(g)
Hydrogen	4(23)	5(4)	5(g)
Recrystallization Temp., C (50% in one hour)	1010-1035(11)	1170-1220(4)	1200-1315(f)
Ductile-Brittle Transition Temperature, C[d]	<-75(5)	-42(4)	-
Tensile Properties[d]			
25 C			
UTS, ksi	127(15)	128(4)	110(e)
YS, 0.2% Offset, ksi	102(14)	104(4)	95(e)
Elongation, %	10(14)	15(4)	22(e)
980 C			
UTS, ksi	55(6)	-	-
YS, 0.2% Offset, ksi	40(6)	-	-
Elongation, %	10(6)	-	-
1095 C			
UTS, ksi	18(1)	-	58(e)
YS, 0.2% Offset, ksi	14(1)	-	52(e)
Elongation, %	31(1)	-	11(e)
1200 C			
UTS, ksi	12(2)	33(4)	45(e)
YS, 0.2% Offset, ksi	6(2)	25(4)	42(e)
Elongation, %	31(2)	13(4)	15(e)
1315 C			
UTS, ksi	-	-	20(f)-30(e)
YS, 0.2% Offset, ksi	-	-	10(f)
Elongation, %	-	-	20(f)

a As-rolled.
b Numbers in parentheses indicate the number of sheets evaluated to determine values.
c Stress-relieved.
d Longitudinal properties.
e Levy, A. V., "Use of Refractory Metals in Air-Breathing Engines", Refractory Metals and Alloys, Interscience Publishers, 1961, p. 610-611.
f Semchyshen, M., McArdle, G.D., and Barr, R.Q., Development of Molybdenum-Base Alloys, Climax Molybdenum Company of Michigan, WADC Technical Report 59-280, October, 1959, page 83, specimens 1597, 1622, and 2527.
g Climax Molybdenum Company of Michigan, Specification CMX-WB-T-1 for Climelt Molybdenum -0.5 per cent Titanium Wrought Bars.

Typical microstructures of the two base materials appear in Figure 1. Ultimate tensile strengths and tensile elongations are plotted as functions of temperature in Figure 2. The high strength of arc-cast Mo-0.5Ti is obtained at the expense of ductility. The recrystallization curves are in Figure 3. Arc-cast Mo-0.5Ti sheet is about 50% recrystallized after one hour at 1200 C, whereas powder-metallurgical molybdenum sheet is about 50% recrystallized after one hour at 1020 C.

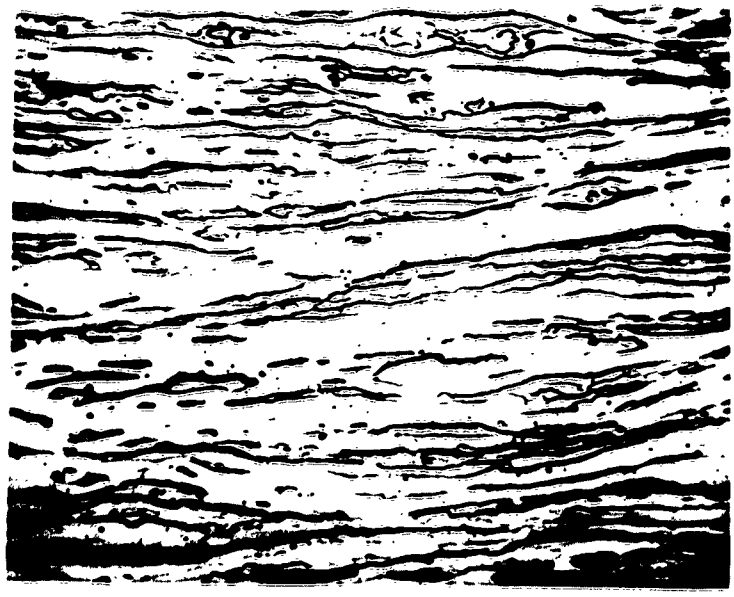
3.0 ALLOY SCREENING

Varying compositions, listed in Table III, of 15 alloy systems were investigated. All sheets which could be fabricated were evaluated at 40 mils, usually in the as-rolled condition. The criteria for screening the alloys were rollability, tensile strength at 1200 C, and ductile-brittle transition temperature.

The selection of trial compositions followed two basic ideas. Initially, we attempted to translate to sheet our experience obtained from developing dispersion-strengthened molybdenum and tungsten wire. This was done by adding various amounts of TiO_2, ZrO_2, Al_2O_3, ThO_2, and Cr_2O_3 to either the molybdenum powder or the molybdic oxide, which was then reduced to molybdenum powder. A billet was then pressed from each type of powder, sintered in hydrogen at 1800 C, and rolled to

MIRCOSTRUCTURE OF BASE MATERIALS

POWDER-METALLURGICAL MOLYBDENUM



ARC-CAST Mo-0.5 Ti

Fig. 1. Microstructure of 40-mil sheets of powder-metallurgical molybdenum and arc-cast Mo-0.5Ti. Photomicrographs are longitudinal at 1000X.

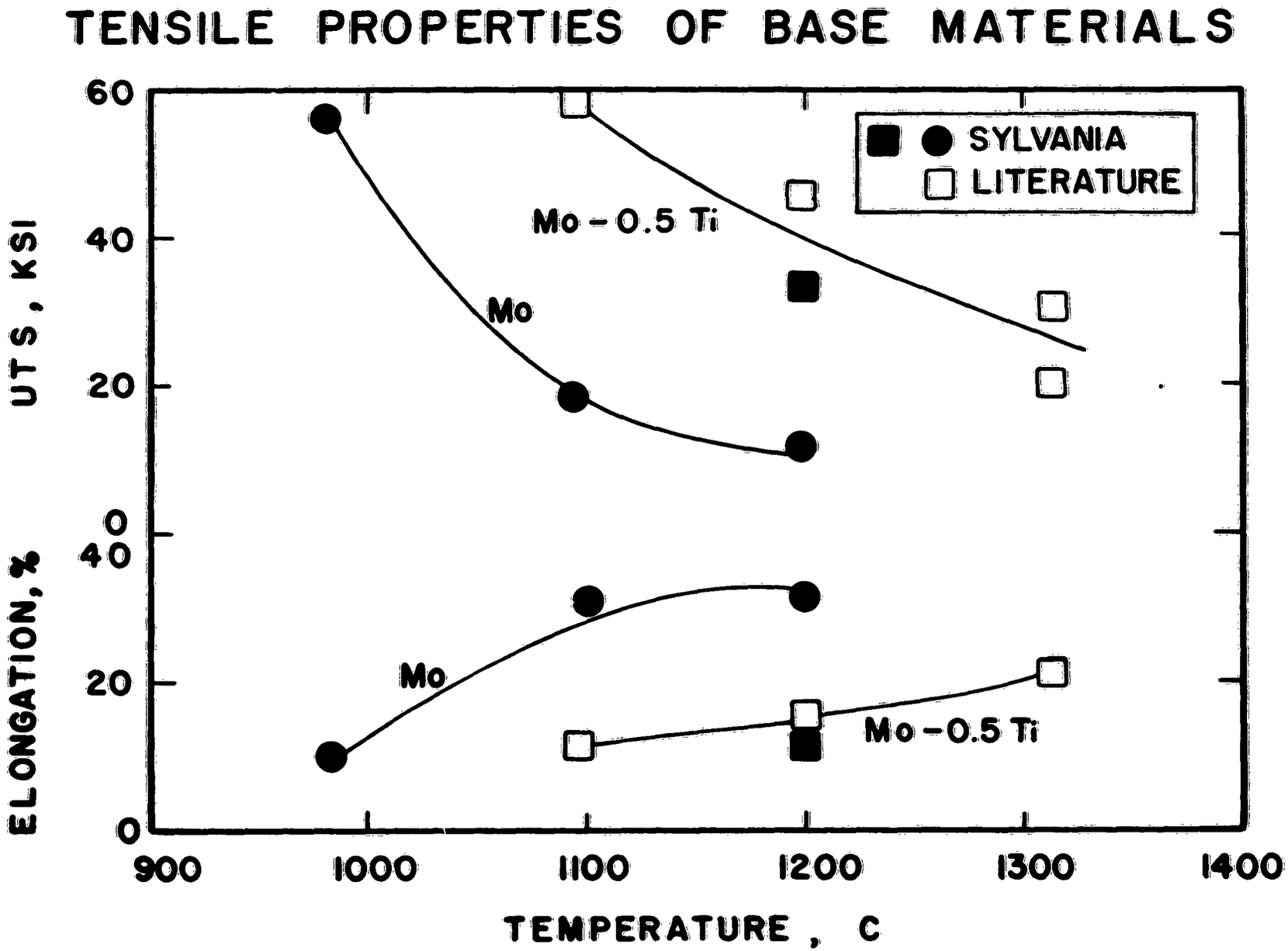


Fig. 2. High-temperature tensile properties of 40-mil sheets of powder-metallurgical molybdenum and arc-cast Mo-0.5Ti.

RECRYSTALLIZATION CURVES FOR BASE MATERIALS

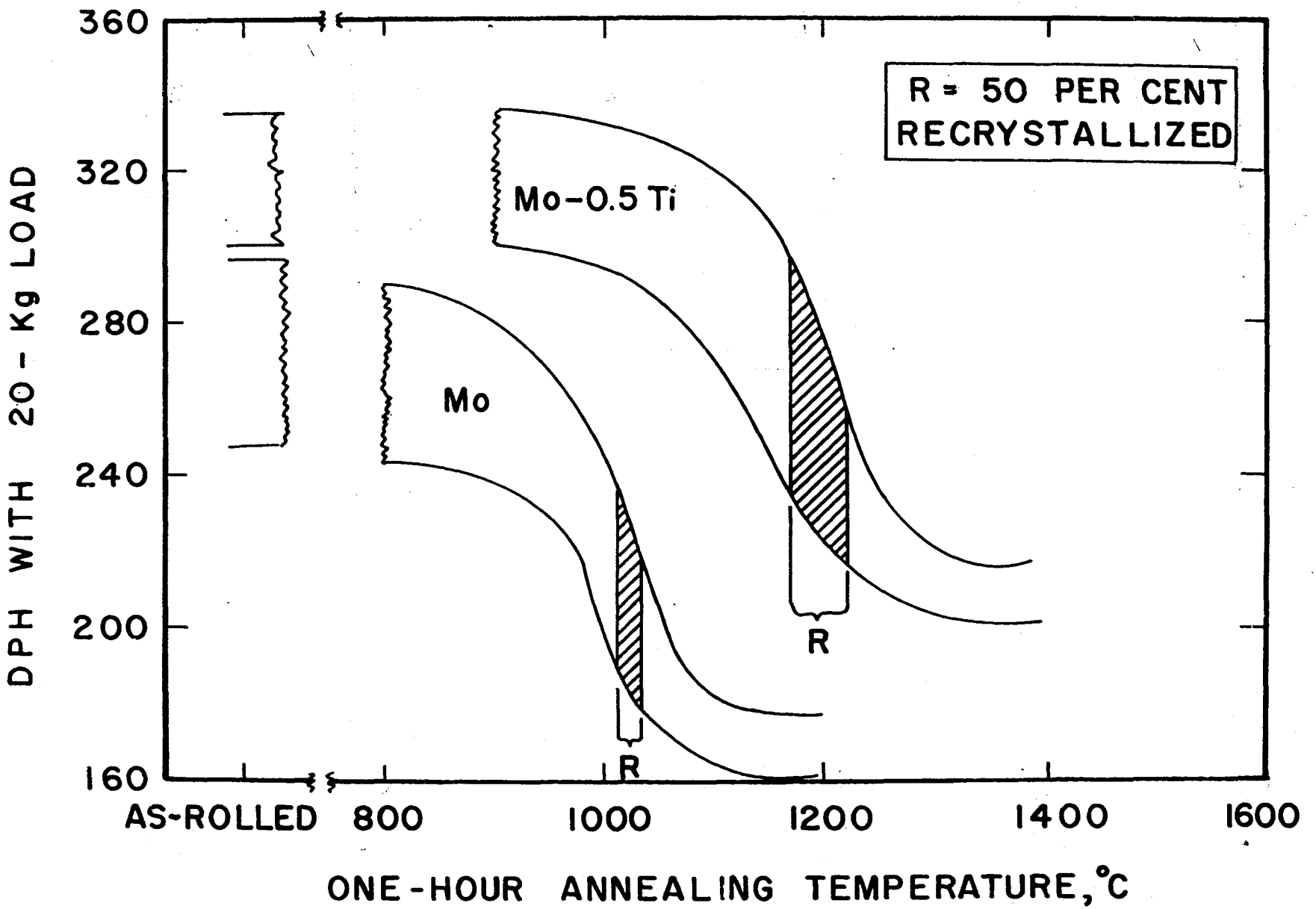


Fig. 3. Recrystallization temperature of 40-mil sheets of powder-metallurgical molybdenum and arc-cast Mo-0.5Ti as determined by hardness measurements.

TABLE III

ALLOY SYSTEMS INVESTIGATED

Alloy System	Nominal Powder Compositions	Interim Report Reference 1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17
$Mo-TiO_2$	$Mo-(0.05,0.25,0.5,1.0)TiO_2$	X	X	X	X	X	X	X	X			X						
	$Mo-0.5TiO_2-0.05C$				X	X												
$Mo-ZrO_2$	$Mo-(0.05,0.25,0.5)ZrO_2$	X	X	X	X	X	X	X										
$Mo-Al_2O_3$	$Mo-(0.05,0.25,0.5)Al_2O_3$	X	X	X	X	X	X	X										
$Mo-ThO_2$	$Mo-(0.5,1.0,2.0)ThO_2$			X	X													
$Mo-Cr_2O_3$	$Mo-(0.5,1.0,2.0)Cr_2O_3$			X	X	X	X	X										
Mo-C	Mo-(0.05,0.1,0.5)C					X	X								X			
Mo-Zr	$Mo-0.5ZrH_2$						X											
Mo-Ti	Mo-(0.5,0.75,1.5,3.0)Ti			X	X	X	X	X				X			X			
	$Mo-0.5TiH_2$						X		X			X						
Mo-W	Mo-30W					X	X	X										
$Mo-W-ZrO_2$	$Mo-25W-0.11ZrO_2-(0.0,0.05)C$						X	X										
Mo-Ti-C	Mo-0.5Ti-(0.05-0.3)C				X	X	X	X	X			X			X		X	
	Mo-0.75Ti-(0.10-0.24)C								X			X						
	Mo-3.2Ti-0.5C					X	X											
	$Mo-0.25TiH_2-0.09C$																X	X
	$Mo-0.5TiH_2-(0.015-0.11)C$																X	X
	$Mo-1.0TiH_2-0.08C$																X	X
	$Mo-1.5TiH_2-0.08C$																X	X
	$Mo-(0.31,0.62)TiC$																X	X
$Mo-Ti-ZrO_2$	$Mo-0.5Ti-0.05ZrO_2$			X	X	X	X	X										
Mo-Ti-Zr	$Mo-0.5Ti-0.07ZrH_2$				X	X	X											
Mo-Nb-C	Mo-1.45Nb-0.25C					X	X	X										
Mo-Ti-Zr-C	$Mo-0.5Ti-0.05ZrO_2-0.05C$					X	X											
	$Mo-0.5TiH_2-0.08ZrH_2-0.09C$																X	
	$Mo-1.27Ti-0.29ZrH_2-0.3C$								X			X						

40-mil sheet for evaluation. The Mo-ThO_2 billets failed during rolling. Though the physical character of most of the alloys was similar to that of dispersion-strengthened wire, the magnitudes of the increases in high-temperature properties fell far short of those obtained in wire.

The oxide concentrations, within the ranges we investigated, that imparted the best high-temperature properties to molybdenum were 1.0% Cr_2O_3, 0.5%TiO_2, and 0.5%ZrO_2. The additions were made in the following ways. Chromic oxide was dissolved in nitric acid and zirconium nitrate was dissolved in water. Each solution was then added to a separate water slurry of MoO_2 and evaporated to dryness. The calcined products were then reduced in hydrogen to the respective powders. Titania powder was simply dry-mixed with molybdenum powder to obtain the Mo-TiO_2 compositions.

The test data for these three alloys appear in Table IV. The 1200 C ultimate tensile strengths, the ductile-brittle transition temperatures, and the recrystallization temperatures are between those respective values for unalloyed molybdenum and arc-cast Mo-0.5Ti. The Mo-1.0%Cr_2O_3 alloy has the highest ultimate tensile strength, 18 ksi.

Our second approach involved the preparation and evaluation of alloys that duplicated the compositions of several arc-cast

TABLE IV

EVALUATION OF BEST MOLYBDENUM - METAL OXIDE SHEETS[a]

Property	Alloy			Base Materials	
	$Mo-1.0Cr_2O_3$	$Mo-0.5TiO_2$	$Mo-0.5ZrO_2$	Powder-Met. Mo	Arc-Cast Mo-0.5Ti
Sheet No.	10-18	7-C3	10-15	-	-
Rollability	Good	Good	Good	Good	-
1200 C Tensile Properties[b]					
UTS, ksi	18	16	14	12	33
YS, 0.2% Offset, ksi	9	8	11	6	25
Elongation, %	24	33	25	31	13
25 C Tensile Properties[b]					
UTS, ksi	135	110	114	127	128
YS, 0.2% Offset, ksi	127	96	103	102	104
Elongation, %	6	12	12	10	15
Ductile-Brittle Transition Temperature, C[b]	-50	-50	-50	<-75	-42
Recrystallization Temperature, C (50% in one hour)	1100	1100	1100	1020	1200

a 40-mil, as-rolled.

b Longitudinal Properties.

molybdenum alloys. This approach, which was ultimately very successful, included the following powder compositions.

(1) Mo-0.5Ti

(2) Mo-0.5Ti-0.05C

(3) Mo-0.5Ti-0.084ZrH_2-0.08C

(4) Mo-1.27Ti-0.29ZrH_2-0.4C

(5) Mo-1.45Nb-0.25C

(6) Mo-30W

(7) Mo-25W-0.11ZrO_2-0.05C

The first five compositions were prepared by dry-mixing the constituent powders. The sixth composition was prepared by co-reducing a mixture of molybdenum and tungsten oxides, while the seventh was made by a co-precipitation technique. During hydrogen sintering, hydrogen evolved from the ZrH_2 and the carbon content decreased.

The rollability of these compositions varied. Billets from compositions (6) and (7) rolled well, those from (1) and (2) rolled fairly well, and those from (4) and (5) rolled poorly. No sheet could be produced from billets of composition (3).

The microstructures of compositions (1), (2), (4), and (5) contained agglomerates, presumably reactive-metal oxides and carbides. The structure of the Mo-30W alloy was a solid

solution of molybdenum and tungsten, while that of Mo-25W-0.11ZrO_2-0.05C was a solid solution of molybdenum and tungsten containing dispersed ZrO_2. The carbon was probably in solid solution with the molybdenum and tungsten.

The ductile-brittle transition temperatures and the tensile properties at 1200 C are compared in Table V to those of their arc-cast counterparts. The ductile-brittle transition temperatures of the sheets from compositions (2), (4) and (5) were greater than 250 C while those of Mo-30W and Mo-25W-0.11ZrO_2-0.04C were -50 C and 12 C respectively.

The 1200 C ultimate tensile strengths range from 28 ksi for Mo-0.5Ti to 56 ksi for Mo-1.27Ti-0.29Zr-0.3C. All but the Mo-0.5Ti alloy (no carbon) have higher 1200 C strengths than that of arc-cast Mo-0.5Ti-C. However, only the powder-metallurgical Mo-0.50Ti-0.043C alloy was stronger than its arc-cast counterpart. Therefore, subsequent investigations involved molybdenum alloyed with titanium and carbon.

4.0 CHARACTERIZATION OF POWDER-METALLURGICAL Mo-Ti-C

This section of the report summarizes a literature survey and many experiments conducted to determine the effects of process and compositional variations on the character of

TABLE V

PROPERTIES OF POWDER-METALLURGICAL AND ARC-CAST ALLOYS

Sheet Composition[c]	Process	Rollability	1200 C Properties			DBTT
			UTS ksi	YS 0.2% Offset ksi	Elong. %	Long. C
Mo (Base)	Powder-Met.	Good	12	6	31	<-75
Mo-0.5Ti	Powder-Met.	Fair	28	22	9	-
Mo-0.5Ti-0.043C	Powder-Met.	Fair	42	33	5	>250
Mo-0.5Ti-0.028C (Base)	Arc-cast	-	33	25	13	-42
Mo-0.5Ti-0.084Zr-0.06C	Powder-Met.	Poor	-	-	-	-
Mo-0.5Ti-0.090Zr-0.02C (TZM)(a)	Arc-cast	-	50 min	25 min	5 min	<0
Mo-1.27Ti-0.29Zr-0.3C	Powder-Met.	Poor	56	41	5	>250
Mo-1.27Ti-0.29Zr-0.3C (TZC)(b)	Arc-cast	-	72	-	-	-
Mo-1.57Nb-0.035C	Powder-Met.	Poor	49	38	7	>250
Mo-1.45Nb-0.25C(b)	Arc-cast	-	77	-	-	-
Mo-30W	Powder-Met.	Good	37	17	9	-50
Mo-30W-0.02C	Arc-cast	-	61	-	-	-
Mo-25W-0.11ZrO_2-0.04C	Powder-Met.	Good	44	29	10	12
Mo-25W-0.11Zr-0.05C(b)	Arc-cast	-	82	-	-	-

a Climax Molybdenum Company of Michigan, Specification CMX-S-TZM-1 for Climelt TZM Sheet, June, 1962.

b Semchyshen, M., McArdle, G. D., Barr, R. Q., Development of Molybdenum-Base Alloys, Climax Molybdenum Company of Michigan, WADC Technical Report 59-280, October, 1959. UTS values determined by linear interpolation of reported tensile values on stress-relieved specimens at 980 and 1315 C.

c The composition of the sheet is assumed to differ from the composition of the mix only in the carbon content. The table gives the carbon content determined by analysis.

powder-metallurgical molybdenum sheet dilutely alloyed with titanium and carbon. The results led ultimately to the selection of Mo-0.5Ti-0.03, which has properties that compare well with those of arc-cast Mo-0.5Ti.

Sintering conditions, titanium and carbon concentrations, in-process thermal treatments, and rolling parameters were investigated.

4.1 Effects of Sintering Conditions

The originally prepared powder-metallurgical Mo-0.5Ti-0.043C sheet (Table V) had an ultimate tensile strength of 42 ksi at 1200 C compared to 33 ksi for the arc-cast Mo-0.5Ti-0.028C sheet. The powder-metallurgical sheet was rolled from a billet sintered at 1800 C in a hydrogen atmosphere.

To determine the effects of sintering time at temperature, sintering atmosphere, and the amount and chemical form of the titanium addition on the 1200 C tensile properties of 40-mil sheet, billets were prepared in the following manner. Thirty-three rolling billets were isostatically pressed from several powder mixes of molybdenum, titanium or titanium hydride, and carbon. They were then sintered at various temperatures from 1800 to 2300 C. Time at temperature ranged from 7.5 to 22 hours at 1800 C to

0.5 to 6.0 hours at 2300 C. Wet and dry hydrogen, dissociated ammonia, and vacuum were used for sintering atmospheres. Most of the sintered billets were from 89 to 95% dense and were about 1.2-inches thick. Most of them were analyzed for titanium, carbon, oxygen, and nitrogen. They were then rolled to 40-mil sheet by a common schedule.

The rolled sheets were analyzed for carbon, oxygen, and nitrogen, and as-rolled hardness and 1200 C tensile properties were determined. The sintering conditions, compositions, and sheet properties are in Table VI.

From the chemical analyses of the billets and sheets, the material contained the amounts of titanium, carbon, oxygen, and nitrogen shown below.

Element	Average	Range
Titanium, %	0.54	0.48-0.61
Carbon, ppm	380	150-660
Oxygen, ppm	98	39-270
Nitrogen, ppm	130	12-630

The room-temperature diamond pyramid hardness averaged 325 and ranged from 273 to 367.

TABLE VI

PROPERTIES OF 40-MIL AS-ROLLED Mo-0.5Ti-C SHEET AS A FUNCTION OF SINTERING TEMPERATURE

Sheet Number	Sintering			Density (b) %	Form of Titanium in Powder Mix	Composition				Hardness DPH 20-kg Load	1200 C Tensile Properties (h)		
	Temp. C	Atmo. (a)	Time hr			Ti (c) %	C (d,e,f) ppm	O (d,e) ppm	N (d,g) ppm		UTS ksi	YS,0.2% Offset ksi	Elong. %
12-3A	1800	H_2*	~7.5	89.7	Ti	0.5	390	100	560	280	36	31	8
12-3B	"	"	"	90.2	"	0.5	370	160	630	273	37	30	7
12-2	"	"	~8	88.5	"	0.5	430	-	-	329	42	33	5
22-18	"	"	22	91.1	TiH_2	0.54	160	150	90	315	47	37	8
Ave.							340	140	430	299	40	33	7
12-K-1	1850	H_2	6	89.8	Ti	0.52	200	270	46	312	50	41	10
12-D-1	"	"	"	91.0	"	0.56	610	76	63	339	60	48	8
12-E-1	"	"	"	86.5	"	0.54	410	190	44	349	57	43	12
22-19	"	"	14	91.7	TiH_2	0.54	220	200	65	317	54	38	8
12-M-1	"	Vac.	6	91.4	Ti	0.50	300	110	19	-	50	42	9
Ave.						0.53	350	170	47	329	54	42	9
22-20	1900	H_2	10	91.7	TiH_2	0.54	180	150	34	323	52	41	8
22-21	1950	H_2	8	91.7	TiH_2	0.54	210	66	53	312	56	43	8
12-D-5A	2000	H_2	3	92.8	Ti	0.56	520	78	23	335	52	41	9
12-E-5	"	"	3	91.8	"	0.50	380	81	37	331	57	46	9
12-D-2	"	"	6	93.0	"	0.52	590	52	34	325	58	46	10
12-E-2	"	"	6	92.6	"	0.55	400	95	30	335	55	39	10
12-M-2	"	Vac.	3	92.5	"	0.49	320	92	32	319	58	-	4
Ave.						0.52	440	80	31	329	56	43	8
12-12	2150	H_2*	1.2	91.2	Ti	0.60	260	39	240	310	52	38	9
12-D-8T	"	H_2	1.5	91.3	"	0.48	650	64	44	325	56	45	7
12-E-8T	"	"	"	93.2	"	0.53	400	86	46	351	49	40	10
12-D-6	"	"	3	93.2	"	0.55	460	84	65	345	54	40	6
12-E-6	"	"	"	93.1	"	0.56	380	100	32	335	55	39	10
12-D-3	"	"	6	94.4	"	0.48	560	66	32	341	59	47	6
12-E-3	"	"	"	94.7	"	0.58	390	76	22	327	55	36	11
12-16	"	"	1.2	90.7	TiH_2	0.54	200	-	330	312	54	38	11
22-26	"	"	3.5	92.7	"	0.54	150	90	18	296	55	40	10
12-3C	"	Dis.NH_3	1.2	91.9	Ti	0.55	460	41	120	327	56	42	11
12-4	"	" "	"	91.2	"	0.55	220	72	630	335	52	36	12
12-8	"	" "	"	90.8	TiH_2	0.61	290	110	570	315	55	34	11
Ave.						0.55	370	75	180	326	54	40	10

12-D-10T	2300	H_2	0.5	92.0	Ti	0.55	660	75	24	325	51	41	8
12-E-10	"	"	"	92.0	"	0.52	550	77	32	358	55	42	12
12-D-4	"	"	6	95.2	"	0.56	620	44	12	367	60	48	10
12-E-4	"	"	"	95.4	"	0.53	270	86	24	321	57	41	9
22-6A	"	"	1.2	92.6	TiH_2	0.54	200	70	47	321	56	43	8
Ave.						0.54	460	70	28	338	56	43	10

a Billets in the 12-E series were sintered in wet hydrogen (dew point range: 11 to 19 C). The other hydrogen-sintered billets were sintered in drier hydrogen (dew point range: -36 to -50 C).

b Sintering rates affected primarily by temperature and time at temperature and somewhat by carbon level in mix, Ti vs. TiH_2, pressing pressure, and sintering atmosphere.

c One and two significant figures indicate nominal addition to powder mix and sintered billet analysis, respectively.

d Values are averages of sintered billet and rolled-sheet analyses.

e Affected by hydrogen dew point and carbon in powder mix.

f Affected by pressing pressure.

g Values >90 ppm attributed to sintering in either dissociated NH_3 or in hydrogen contaminated with dissociated NH_3.

h Longitudinal.

* Indicates that the hydrogen contained some dissociated NH_3.

The 1200 C tensile properties were as shown below.

Property	Average	Range
UTS, ksi	53	36-60
YS (0.2% offset), ksi	40	30-48
Elongation, %	9	5-12

The most significant observation from this work is that the 1200 C strengths of sheets processed by sintering at 1800 C are significantly lower than the 1200 C strengths of those processed by sintering at 1850 to 2300 C.

Sintering Temp. C	1200 C UTS ksi	1200 C YS, 0.2% Offset ksi
1800	40	33
1850	54	42
1900	52	41
1950	56	43
2000	56	43
2150	54	40
2300	56	43

Ultimate tensile strengths of individual sheets are plotted as a function of sintering temperature in Figure 4 which shows that sintering at or above 1850 C consistently results in sheets with 1200 C ultimate tensile strengths ranging from 49 to 60 ksi.

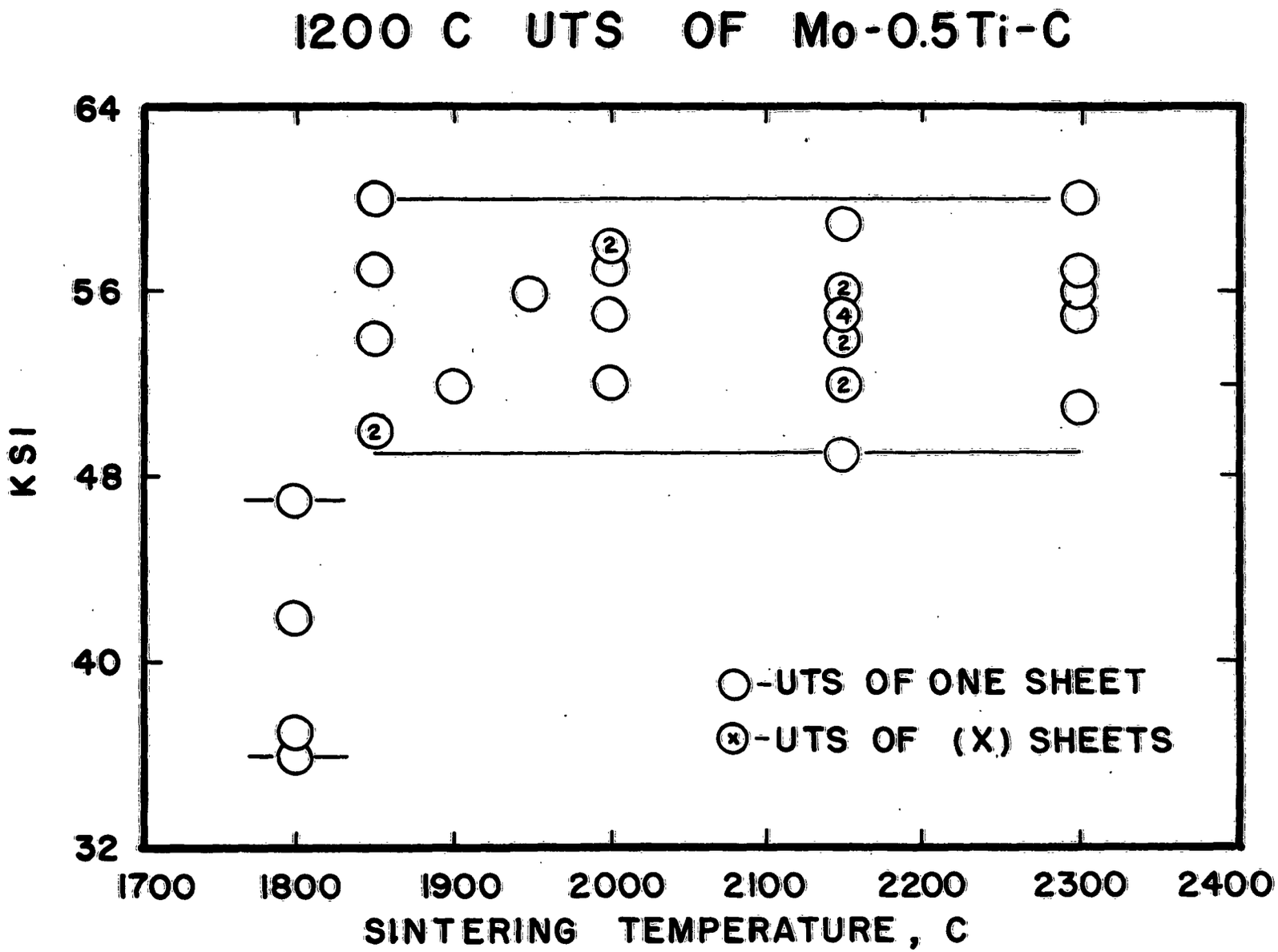


Fig. 4. Effect of sintering temperature on the ultimate tensile strength of 40-mil sheets of powder-metallurgical Mo-0.5Ti containing 0.015 to 0.066% carbon.

The room-temperature hardness reflects this dependence on sintering temperature to a lesser degree than does the 1200 C strength.

Carbon, oxygen, and nitrogen contents were affected to various degrees by the imposed variations in sintering conditions, but not to an extent to influence the hardness and 1200 C strength properties. Materials processed by sintering in dry hydrogen or in vacuum have the lowest oxygen and nitrogen contents. Material processed by sintering in wet hydrogen are relatively high in oxygen, and material processed by sintering in dissociated ammonia or dry hydrogen contaminated with dissociated ammonia were relatively high in nitrogen. Sheets made from mixes of molybdenum and carbon with either titanium or titanium hydride powders have similar properties.

In subsequent experiments, 2300 C was frequently used as the sintering temperature because adequate densities could be obtained in a relatively short time.

4.2 Effects of Carbon Content

Our first work (Table V) indicated that the high-temperature strength of Mo-Ti-C alloys is dependent on the carbon content. To investigate this effect several

rolling billets were isostatically pressed from several powder mixes of molybdenum, titanium or titanium hydride, and variable amounts of carbon. The billets were sintered at various temperatures from 1850 to 2300 C, the temperature range used to process sheet with consistently high strength at 1200 C. Dry hydrogen, dissociated ammonia, and vacuum were used for sintering atmospheres. The billets were analyzed chemically and rolled to 40-mil sheet as in the previous experiment. The sheets were evaluated for composition, room-temperature hardness, and 1200 C tensile properties. The sintering conditions, compositions, and sheet properties are listed in Table VII according to increasing carbon content. Those sheets in the previous experiment which were processed by sintering at 1850 to 2300 C are also listed in the same manner, making a total of 44 sheets. Carbon contents range from 20 to 660 ppm.

The most significant observation from these data is that uniformly high strengths at 1200 C are consistently obtained for sheets containing at least 200 ppm of carbon. The ultimate tensile and yield strengths of the individual sheets are plotted as a function of carbon content in Figures 5 and 6, respectively. Figure 5 shows that the ultimate tensile strength increases from

TABLE VII

PROPERTIES OF 40-MIL AS-ROLLED Mo-0.5Ti-C SHEET[a] AS A FUNCTION OF CARBON CONTENT

Sheet Number	Composition (b) Carbon ppm	(b) Oxygen ppm	(b,c) Nitrogen ppm	(d) Titanium %	Sintering Temp. C	Atmo.[e]	Hardness DPH 20-kg Load	1200 C Tensile Properties[f] UTS ksi	YS 0.2% Offset ksi	Elong. %
12-H-1	20	640	22	0.50	1850	H_2	277	26	21	9
12-H-3	22	650	20	0.49	2150	"	288	28	22	8
5-1	23	380	37	0.5	2300	"	244	26	20	10
12-10	30	130	220	0.53	2150	dis.NH_3	275	31	23	12
4-1	34	54	14	0.5	2300	H_2	267	24	18	18
13-1	37	100	24	0.49	2300	"	267	37	29	10
12-17	70	130	31	0.49	2150	"	256	26	20	10
12-9	70	100	68	0.55	2150	dis.NH_3	258	25	20	11
11-1	76	180	8	0.5	2300	H_2	271	36	20	12
12-18	80	90	100	0.55	2150	H_2*	273	30	24	8
12-K-2	110	100	14	0.56	2150	H_2	289	42	35	9
22-14B	130	110	20	0.54	2300	"	280	49	38	7
2-1	140	56	16	0.5	2300	"	308	58	41	9
22-26	150	90	18	0.54	2150	"	296	55	40	10
22-14A	170	110	16	0.54	2300	"	371	48	37	17
22-20	180	150	34	0.54	1900	"	323	52	41	8
12-K-1	200	270	46	0.52	1850	"	312	50	41	10
22-6A	200	70	47	0.54	2300	"	321	56	43	8
12-16	200	-	330	0.54	2150	H_2*	312	54	38	11
22-21	210	66	53	0.54	1950	H_2	312	56	43	8
12-4	220	72	630	0.55	2150	dis.NH_3	335	52	36	12
22-19	220	200	65	0.54	1850	H_2	317	54	38	8
12-12	260	39	240	0.60	2150	H_2*	310	52	38	9
12-E-4	270	86	24	0.53	2300	H_2	321	57	41	9
12-8	290	110	570	0.61	2150	H_2*	315	55	34	11
12-M-1	300	110	19	0.50	1850	Vac.	-	50	42	9
12-M-2	320	92	32	0.49	2000	"	319	58	-	4
12-E-6	380	100	32	0.56	2150	H_2	335	55	39	10
12-E-5	380	81	37	0.50	2000	"	331	57	46	9
12-E-3	390	76	22	0.58	2150	"	327	55	36	11
12-E-2	400	95	30	0.55	2000	"	335	55	39	10
12-E-8T	400	86	46	0.53	2150	"	351	49	40	10
12-E-1	410	190	44	0.54	1850	"	349	57	43	12
12-D-6	460	84	65	0.55	2150	"	345	54	40	6
12-3C	460	41	120	0.55	2150	dis.NH_3	327	56	42	11
12-D-5A	520	78	23	0.56	2000	H_2	335	52	41	9

12-E-10	550	77	32	0.52	2300	H_2	358	55	42	12
1-1	560	52	22	0.5	2300	"	339	61	44	8
12-D-3	560	66	32	0.48	2150	"	341	59	47	6
12-D-2	590	52	34	0.52	2000	"	325	58	46	10
12-D-1	610	76	63	0.56	1850	"	339	60	48	8
12-D-4	620	44	12	0.56	2300	"	367	60	48	10
12-D-8T	650	64	44	0.48	2150	"	325	56	45	7
12-D-10T	660	75	24	0.55	2300	"	325	51	41	8

a Processed by sintering at or above 1850 C.

b Values are averages of sintered billet and rolled sheet analyses.

c Values >90 ppm attributed to sintering in either dissociated NH_3 or in H_2 contaminated with dissociated NH_3.

d One and two significant figures indicate nominal addition to powder mix and sintered billet analysis, respectively.

e Billets in the 12-E series were sintered in wet hydrogen (dew point range: 11 to 19 C). The other hydrogen-sintered billets were sintered in drier hydrogen (dew point range: -36 to -50 C).

f Longitudinal.

* Indicates that the hydrogen contained some dissociated ammonia.

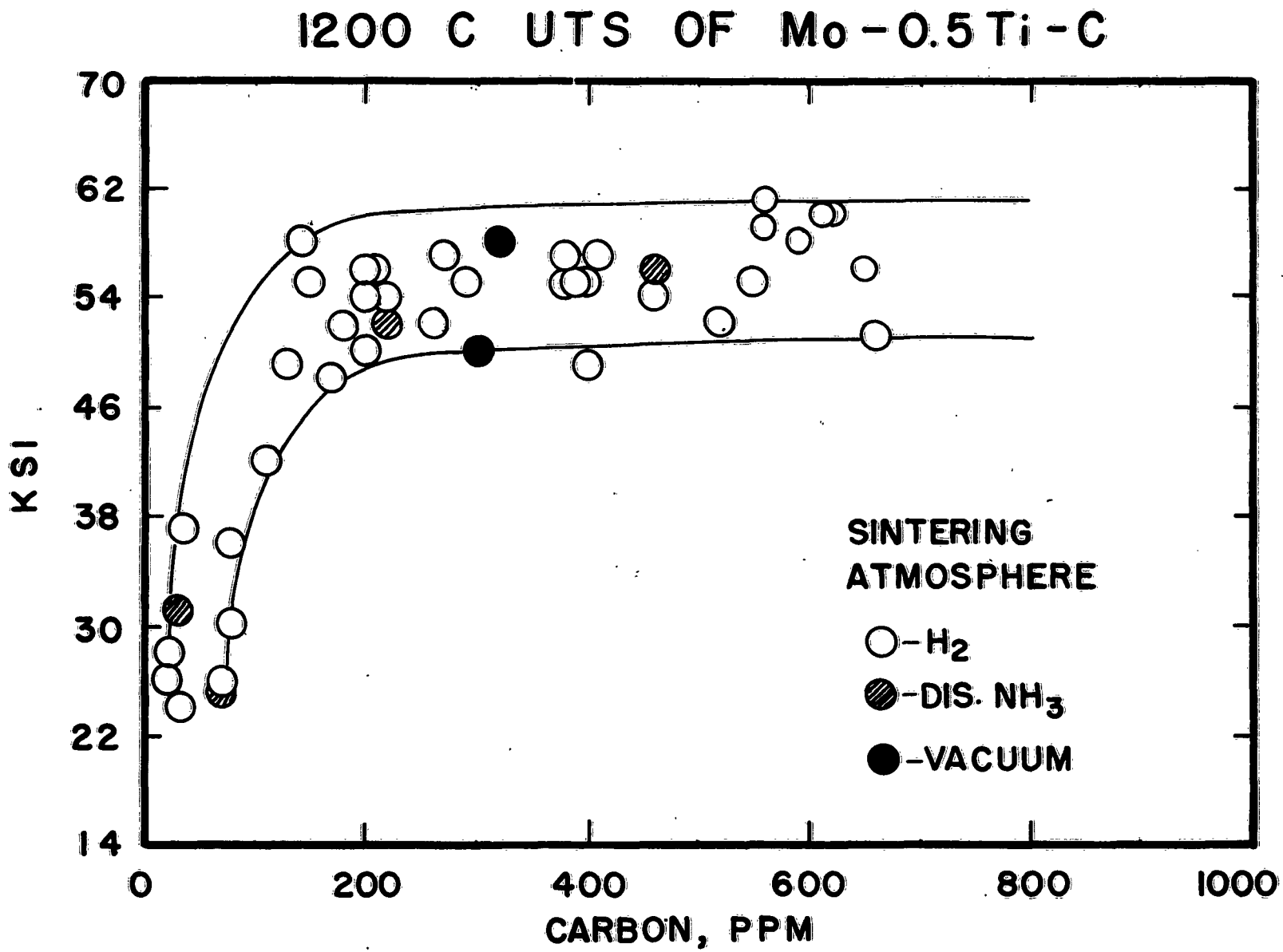


Fig. 5. Effect of carbon content on the 1200 C ultimate tensile strength of 40-mil sheets of powder-metallurgical Mo-0.5Ti rolled from billets sintered at various temperatures from 1850 to 2300 C.

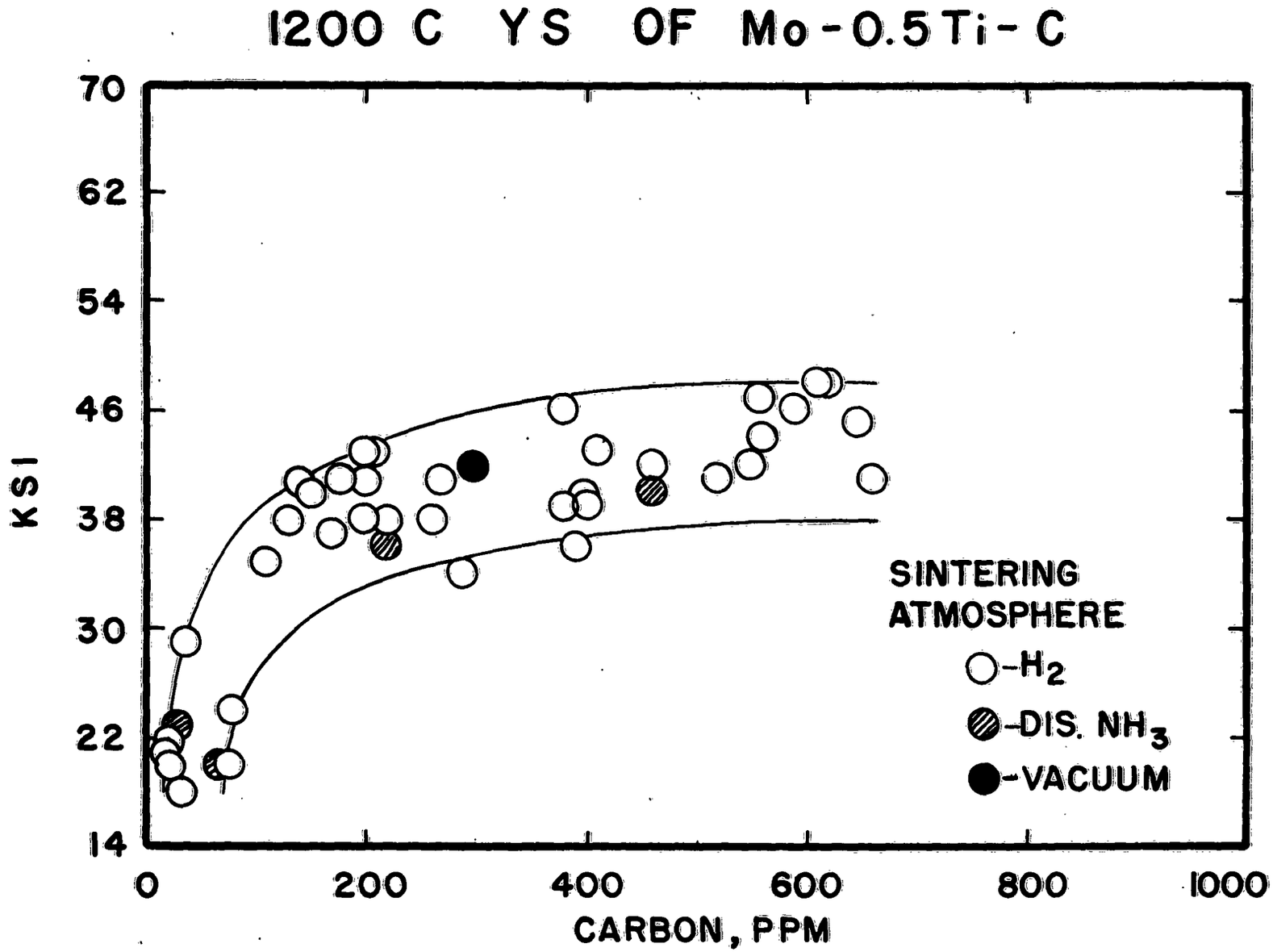


Fig. 6. Effect of carbon content on the 1200 C yield strength (0.2% offset) of 40-mil sheets of powder-metallurgical Mo-0.5Ti rolled from billets sintered at various temperatures from 1850 to 2300 C.

about 24 ksi to a range of 50 to 60 ksi as the carbon content increases from 20 to 200 ppm. Increasing the carbon content from 200 to 660 ppm does not significantly alter the strength. The yield strength follows a similar trend. However, increasing the carbon content from 200 to 660 ppm results in a gradual increase of the average yield strength.

The ultimate tensile- and yield-strength ranges are compared in Figure 7. It is interesting to note that the solubility limit of carbon in molybdenum in the range of sintering temperatures we used is about 200 ppm[1].

Most of the tensile elongations fall in the 6 to 12% range. They are not affected by carbon content. Room-temperature hardness is plotted as a function of carbon content in Figure 8. The hardness increases with increasing carbon.

The materials processed by sintering either in dry hydrogen or in a vacuum have oxygen contents ranging from 44 to 650 ppm. Low oxygen values are usually associated with high carbon contents and vice versa. Apparently, the presence of a sufficient amount of carbon prevents the titanium from oxidizing during the sintering cycle. The nitrogen contents in these same materials range from 8 to 65 ppm. They were not affected by carbon content.

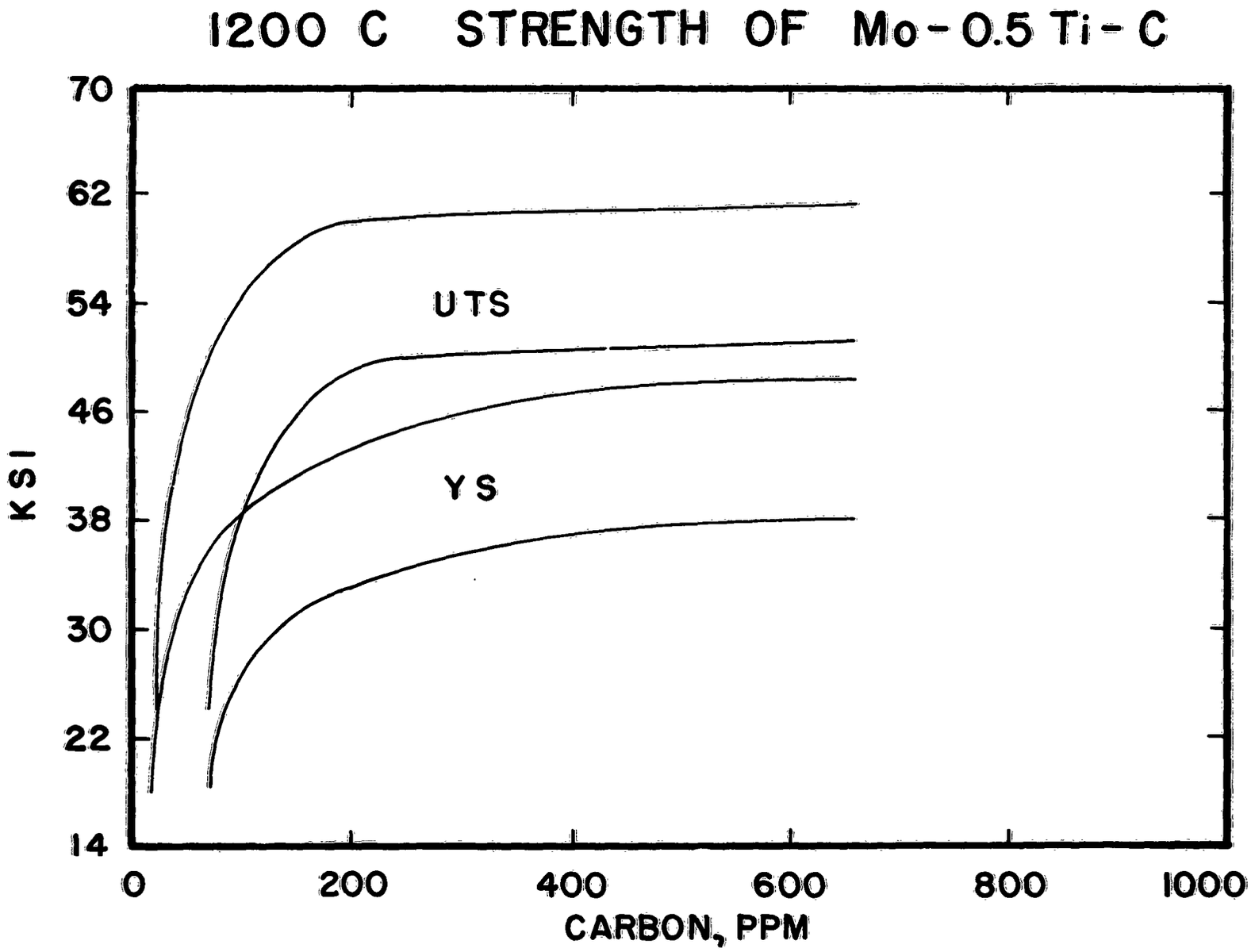


Fig. 7. Effect of carbon content on the ultimate tensile and yield (0.2% offset) strengths at 1200 C of 40-mil sheets of powder-metallurgical Mo-0.5Ti rolled from billets sintered at various temperatures from 1850 to 2300 C.

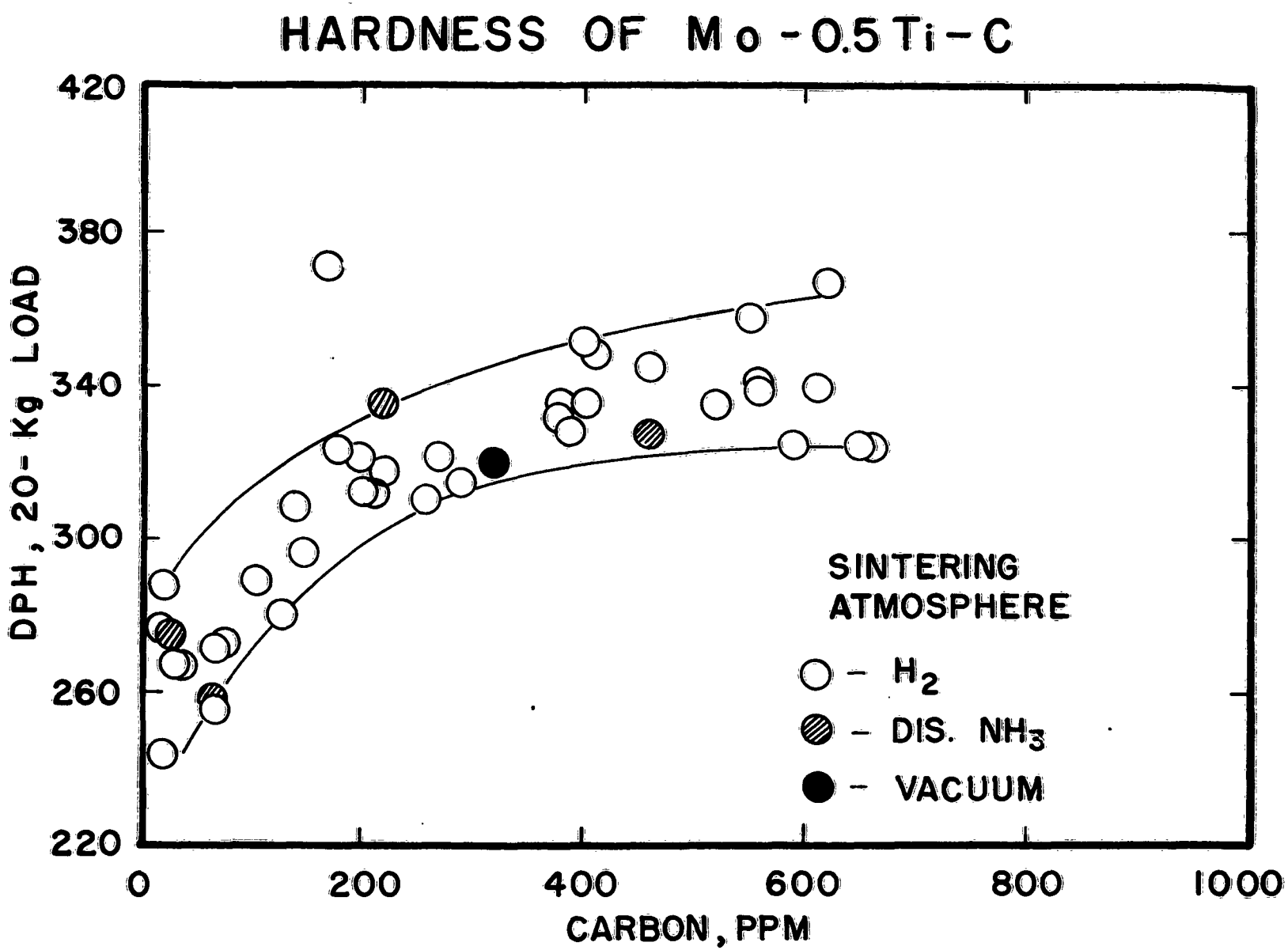


Fig. 8. Effect of carbon content on the room-temperature hardness of 40-mil sheets of powder-metallurgical Mo-0.5Ti rolled from billets sintered at various temperatures from 1850 to 2300 C.

Another significant observation is that sheets containing at least 200 ppm carbon have good strengths regardless of the fact that some of them contain substantial amounts of either oxygen or nitrogen.

4.3 Effects of Titanium Content

The following work was done to determine the effect of variations in titanium content on strength.

Sheets containing 0.33, 0.54, and 0.86% titanium, and at least 200 ppm carbon were processed by sintering at either 2150 or 2300 C.

The results of tensile tests at 1200 C are shown below.

Composition, %		1200 C Tensile Properties		
Titanium	Carbon	UTS, ksi	YS(a), ksi	Elong., %
0.33	0.030	29	23	26
0.54	0.020	56	43	8
0.86	0.027	54	37	11

a 0.2% offset.

A titanium concentration of 0.5% is adequate.

At this stage of our development work, we specified the following conditions for the molybdenum alloy that we developed.

1. Sintering temperature, 1850 to 2300 C.
2. Composition
   - A. Titanium, 0.50 ± 0.05%
   - B. Carbon, 0.03 ± 0.01%

The composition of this powder-metallurgical alloy, designated MTC, is similar to that for the arc-cast alloy, Mo-0.5Ti.

4.4 Strengthening Mechanism in MTC

The sensitivity of the high-temperature strength of MTC to changes in sintering temperature and carbon content clearly showed the need for a knowledge of the strengthening mechanism.

Definition of the mechanism required information on the effects of interstitial elements, in-process heat treatments, and rolling schedule variations on the microstructure, hardness, and high-temperature strength of the alloy. Part of the necessary experimental work was conducted at Towanda[2], and part was subcontracted to the General Telephone and Electronics Laboratories[3,4]. At the latter location a survey of the literature was made on strengthening in arc-cast molybdenum-titanium alloys. The significant portions of that survey are presented below.

4.4.1 Literature Survey

Increased strengths for the Mo-Ti alloys were reported to be dependent on dispersions formed by solid-state reactions between titanium and

the interstitial elements. Bufferd et al[5] and Jaffee[6] found that high-temperature stress-rupture strengths were improved when finely dispersed TiO_2 was present. A dispersion of TiN was formed by Mukherjee and Martin[7] by diffusion of nitrogen into arc-cast Mo-1.0Ti. An investigation of surficial contamination of Mo-0.5Ti sheet at Universal Cyclops[8] indicated that nitrogen raised the recrystallization temperature of the contaminated layer. From strength data on various arc-cast alloys, Semchyshen and Barr[9] concluded that a critical C/Ti ratio is necessary for optimum strengthening of Mo-Ti-C alloys.

The influence of heat treatment and composition on the microstructure and properties of arc-cast Mo-Ti alloys was studied in detail by Chang[10]. Electron metallography, X-ray diffraction, and chemical analyses were used to determine the interrelationships among the reacting phases over a wide temperature range. For Mo-1Ti-0.1Zr-0.14C, hardening was caused by the precipitation of TiC accompanied by dissolution of Mo_2C, which was stable only at high temperatures. Also, the titanium appeared to increase the high-temperature solubility of carbon in the alloy.

For a Mo-0.5Ti-0.035C alloy, Chang found that TiC existed at temperatures below 1650 C. The alloy in the recrystallized form did not show an aging response to heat treatment, but the 1200 C strength of the alloy in the worked form was significantly higher (58-62 ksi) than that normally obtained (37 ksi).[3,10] He suggested that the higher than normal strengths resulted from strain-induced precipitation of TiC during final working after a 1650 C heat treatment. Chang described the alloy as not age-hardenable by thermal treatment alone because of "restricted carbide solutioning at high temperatures" rather than because of any deficiency of carbide.

4.4.2 Effects of Interstitial Elements

Experiments performed by the General Telephone and Electronics Laboratories indicated that large concentrations of nitrogen and oxygen do not significantly influence the 1200 C strength of the powder-metallurgical Mo-Ti-C alloys. Results representative of the bulk of the data are shown in Table VIII. There is considerable

variance in the strength values. No correlation with the changes in any particular interstitial impurity is apparent. The strengths of samples 90 and 92 are higher than those of the others, presumably because of their higher carbon content.

TABLE VIII

EFFECTS OF INTERSTITIAL ELEMENTS ON THE 1200 C TENSILE PROPERTIES OF 40-MIL Mo-Ti-C SHEET[a]

Sheet Number	Carbon ppm	Oxygen ppm	Nitrogen ppm	UTS ksi	Elongation %
		High Oxygen			
3-4	78	2300	100	38	12
37	84	1900	200	24	15
40	56	1100	540	37	21
1-4	29	1500	90	34	10
		High Nitrogen			
90	480	260	930	49	12
92	410	250	930	49	10
		High Oxygen and Nitrogen			
34	67	1300	410	39	7
67	45	660	840	32	26
		Control			
106	58	220	50	42	9
107	73	330	44	43	13

a The sintering temperature was 1700 C. Strength properties would have been higher if sintering had been done at or above 1850 C. The titanium content was 0.5% and no in-process heat treatment was used.

The materials, for which the data are presented in Table VIII, were all processed similarly by presintering at 1000-1100 C in vacuum, sintering one-half hour at 1700 C to a density of about 85%, then canning in molybdenum prior to forging at 1700 C, and rolling to sheet at 1350 C. All but samples 106 and 107 were exposed to a nitrogen-containing atmosphere at temperatures between 1500 and 1700 C.

4.4.3 Titanium Carbide Dispersion

The lack of significant effects from oxygen and nitrogen and the obvious influence of carbon, presented in section 4.2, indicate that carbon is the interstitial element which is active in the strengthening of the MTC alloy. Carbon alone is not sufficient to provide the observed strength increases. This is indicated by a 1200 C tensile strength of 13 ksi, which was determined for a Mo-0.074C alloy as compared with 12 ksi for unalloyed molybdenum.

The indispensable role of titanium found in our investigation and others[8,10], and the large influence of carbon indicate that titanium carbide

is the strengthening agent. The presence of a titanium carbide precipitate was verified by Chang in Mo-1Ti-0.1Zr-0.14C, as mentioned previously. He also showed that molybdenum carbides are stable only at high temperatures, ruling them out as strengtheners at 1200 C.

While the dispersed TiC phase is presumed to be the strengthening agent, the actual mechanism for obtaining the TiC dispersion remains to be determined. Temperature-induced precipitation is not active in forming the strengthening dispersion in the annealed alloy as was demonstrated by Chang and is indicated by the plots of Figure 9 from the G. T. and E. investigation. In the figure, room-temperature hardness is plotted as a function of heat-treatment temperature for powder-metallurgical Mo-0.5Ti-0.08C and Mo-0.5Ti-0.16C and for arc-cast Mo-Ti-Zr-C(10). Only the last composition shows an age-hardening response.

While heat treatment alone does not result in the formation of a precipitate, it is possible that the application of strain in the appropriate temperature range may alter the kinetics of precipitation by providing an increased number of

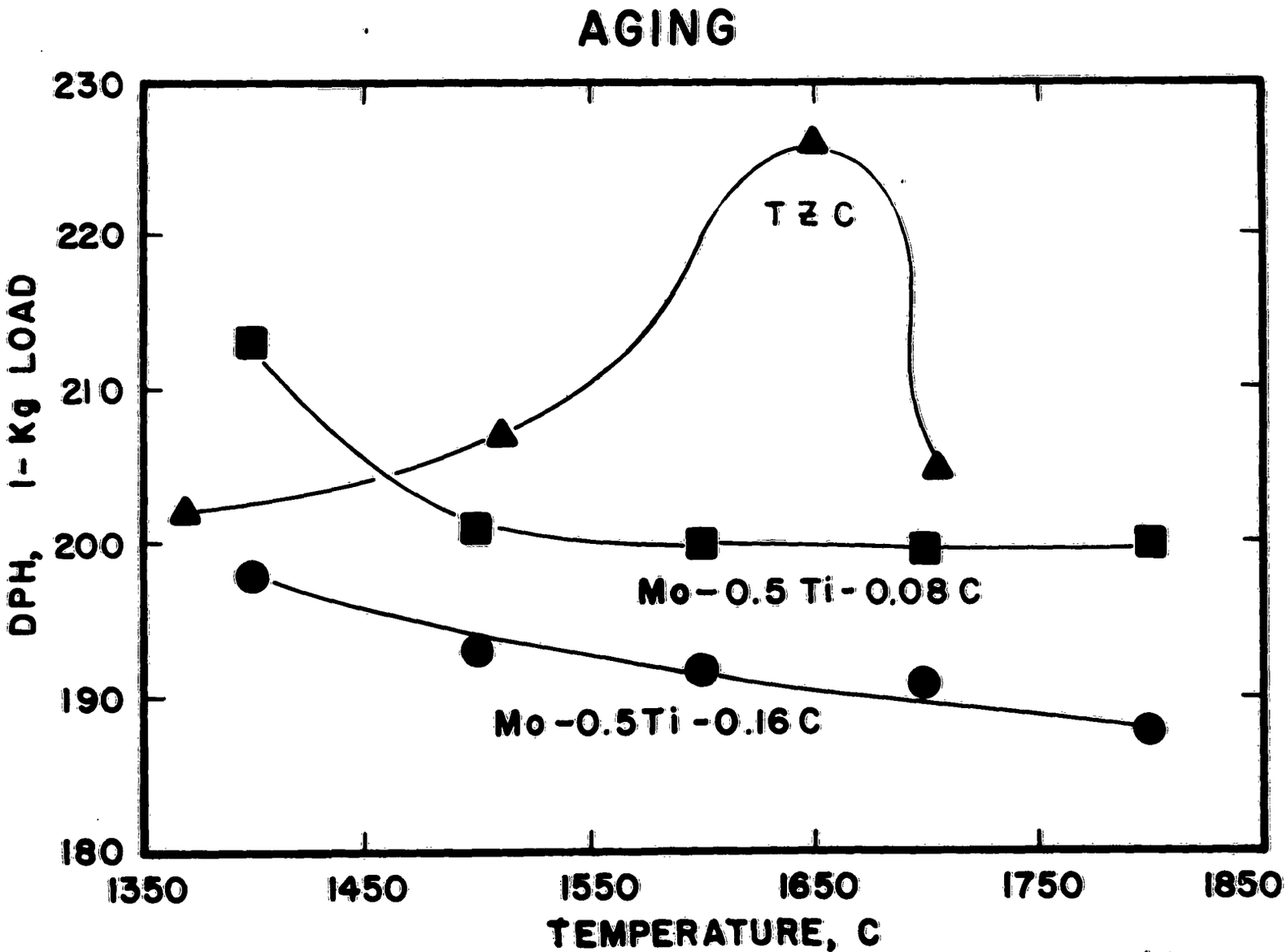


Fig. 9. Room-temperature hardness of recrystallized arc-cast TZC rod[9] and powder-metallurgical Mo-0.5Ti-C sheet[3] after one hour at various temperatures. TZC shows an aging response.

nucleation sites. The rolling operations during processing of MTC sheet probably provide the necessary conditions for such strain-induced precipitation, making possible the formation of a dispersed phase of titanium carbide.

Theoretically, the fineness of a strain-induced precipitate and the resultant matrix strengthening are determined by the amount of strain and the temperature at which strain occurs. Accordingly, sheet properties should be enhanced by the appropriate selection of rolling schedule, rolling temperature, and in-process heat treatment. The experiments described in the following section were designed to test these possibilities.

4.4.4 Heat-Treatment Effects on TiC Strengthening of MTC

The response of MTC rolled sheet to an in-process heat treatment was investigated to determine the effects of aging on strained material and to evaluate the possibilities of enhancing final sheet properties by in-process solutioning of TiC prior to final working(4).

Based on the sintering data presented in section 4.1, MTC billets were sintered at 2300 C. Apparently, sintering at about that temperature

facilitates the dissolving of titanium and carbon by increasing their rate of dissolution and raising their solubilities in the matrix. Also titanium is a liquid above about 1800 C. For the arc-cast alloy the dissolution of titanium and carbon is accomplished during melting, although excessive precipitate growth during cooling and extrusion may require re-solutioning to achieve an optimum dispersion.

One-inch-thick sintered MTC billets were forged to 400 mils at 1500 C; then samples were heat-treated at 1350, 1550, 1700, 1900 and 2150 C. After rolling to 40 mils at 1200 C, the chemical compositions were checked and the tensile properties at 1200 C were measured. The results are shown in Table IX. The high-temperature strengths of the sheets heat-treated at 1700 C and above are obviously improved (67-69 ksi) over that of the control (56 ksi), while heating below 1700 C resulted in lowered strengths (48-50 ksi).

The marked response of the strained sheet to heat treatment of the plate is indicative of strengthening by strain-induced precipitation. Strengths higher than that of the control can be

attributed to dissolution of TiC followed by precipitation during subsequent deformation at a lower temperature (1200 C). Strengths lower than that of the control apparently resulted because TiC did not dissolve in the matrix at 1350 and 1550 C, but instead, coalesced sufficiently to lessen the effectiveness of the dispersion. The improvement in final sheet properties by proper in-process heat treatment, prior to final working, is amply demonstrated by these results.

TABLE IX

EFFECT OF HEAT TREATMENT AT 4CC MILS ON THE 1200 C TENSILE PROPERTIES OF 4C-MIL MTC SHEET

Temperature C	Time at Temp. hr	Carbon %	UTS ksi	YS,0.2% Offset ksi	Elongation %
2150	0.50	0.030	69	64	9
1900	1.0	0.029	67	55	10
1700	2.0	0.031	67	53	12
None (Control)		0.038	56	46	12
1550	7.5	0.037	50	42	11
1350	18	0.038	48	41	11

Figure 10 shows the 1200 C strengths of sheets as a function of carbon content. The sheets were processed with a re-solution heat treatment prior to final working as discussed above. The ultimate-strength range for sheet without the heat treatment is shown for comparison. The samples include those heat-treated at 1700 C and above in the preceding experiment and samples 22-10A, 11A, 12A, which were discussed in Interim Report No. 17.

4.4.5 Effects of Rolling on TiC Strengthening of MTC

Rolling experiments were selected to evaluate the effect of strain on strengthening[2]. Different amounts of strain were introduced into sheet by similarly rolling plates, which were either 620 or 400 mils thick, to 40-mil sheet. Additional variations of strain were introduced by rolling the plates to 120 mils at either 1100 C or 1400 C before rolling to 40 mils at 1100 and about 200 C. Sheets from plates solution-treated for two hours at 1700 C were compared with sheets from untreated plates at comparable strain levels. Tensile properties of

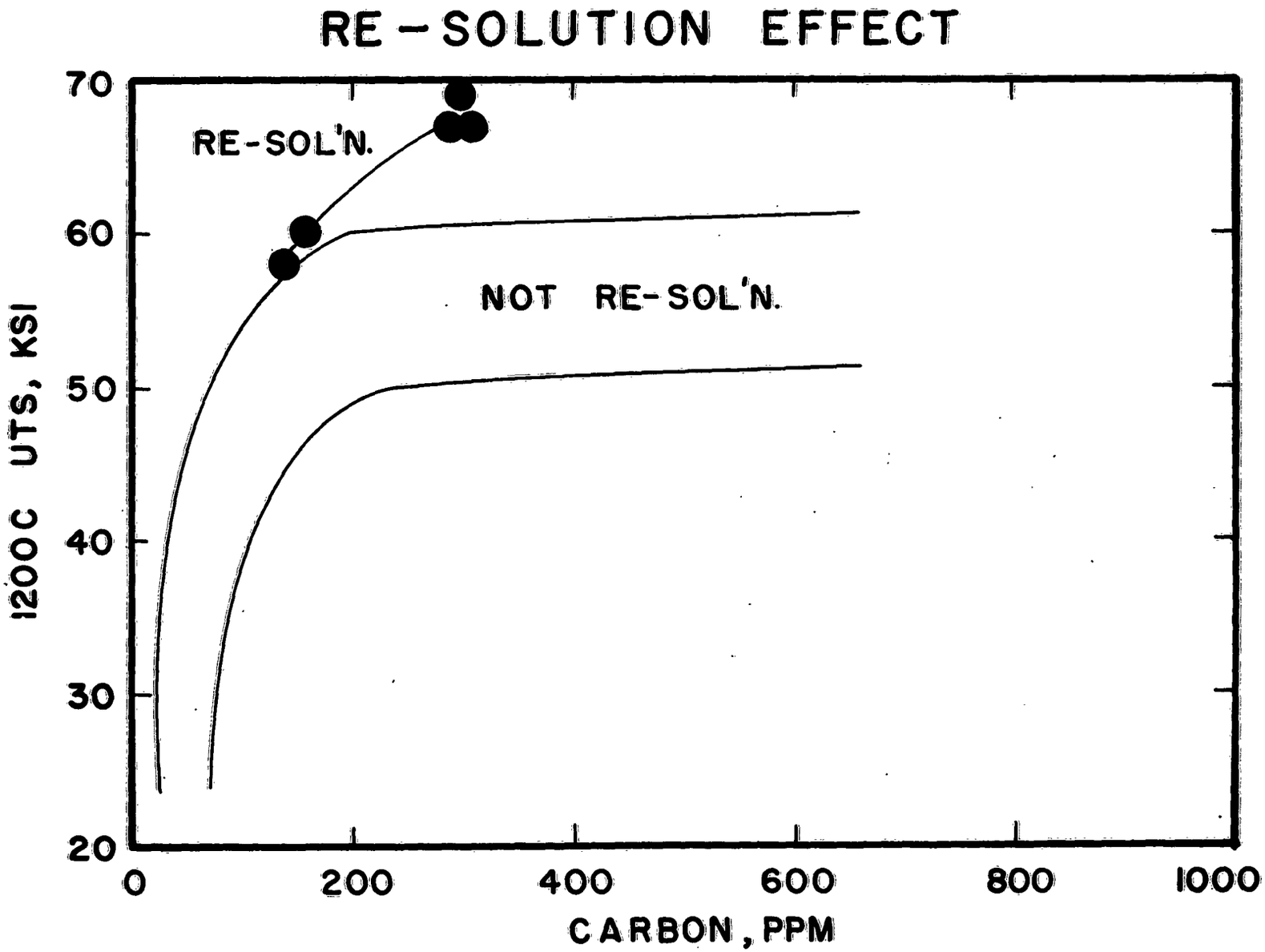


Fig. 10. Effect of re-solution heat treatment of 400-mil MTC plate at or above 1700 C on the 1200 C ultimate tensile strength of 40-mil sheet.[2,4]

the 40-mil sheets were measured at 1200 C and the ultimate tensile strengths are shown in Table X. the above procedure is illustrated schematically in Figure 11.

TABLE X

EFFECT OF HEAT TREATMENT AND STRAIN ON STRENGTH OF 40-MIL MTC SHEET

Heat Treatment[a]			Rolling Temperature[b] C	UTS at 1200 C, ksi Plate Thickness at Heat Treatment	
hrs		Temperature C		620 mils	400 mils
-	None	-	1100	48	56
2		1700	1100	53	60
-	None	-	1400	49	(c)
2		1700	1400	49	52

a This heat treatment was applied to the plate prior to intermediate and final rolling.

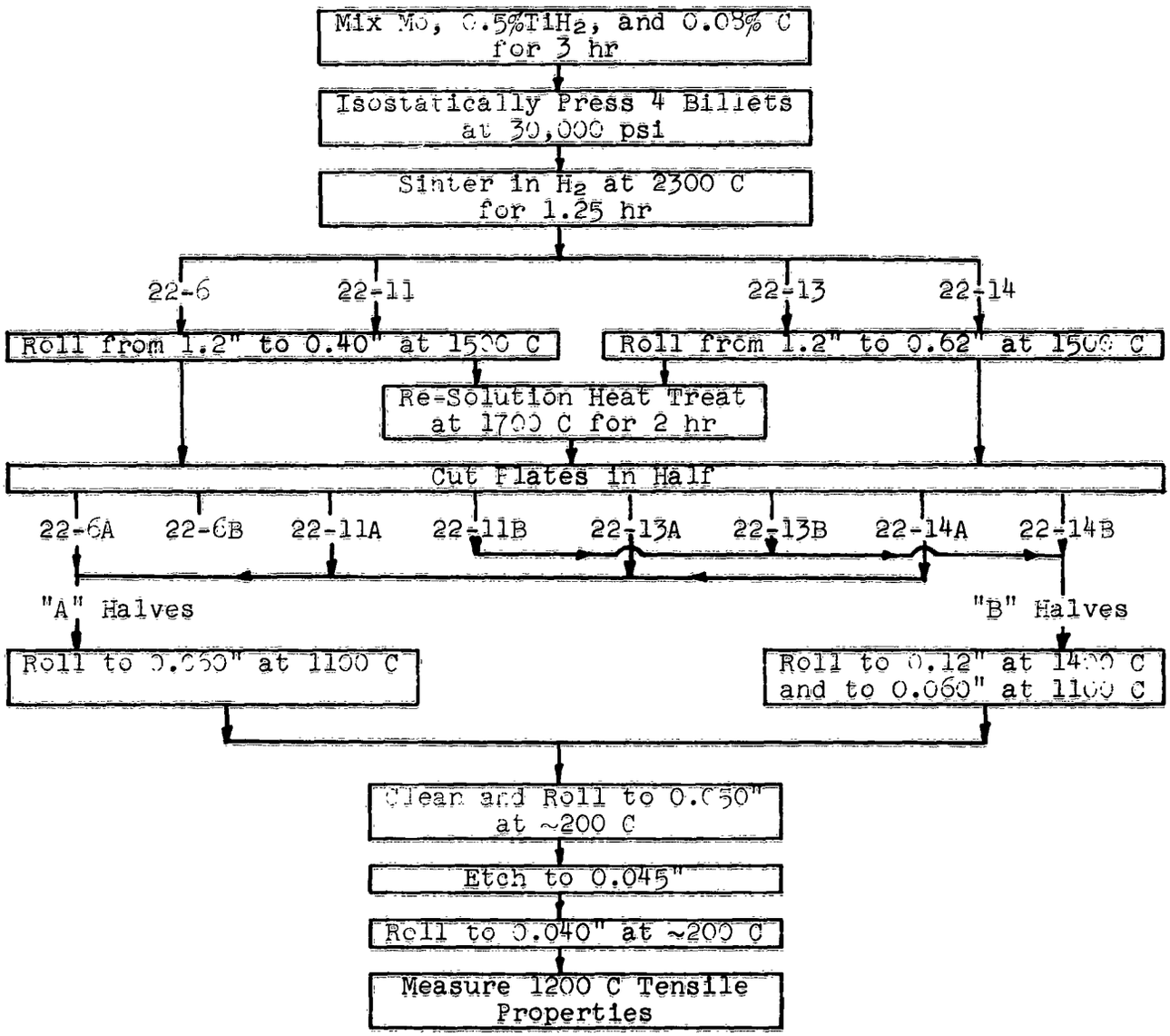
b The material was rolled at this temperature to 120 mils thick, then rolled to 40 mils at 1100 and about 200 C.

c Not rolled to sheet.

The results were again consistent with a strain-induced precipitation mechanism. The highest strength (60 ksi) was obtained for the sheet rolled at 1100 C from the 400-mil plate which was

FIGURE 11

PROCEDURE FOR STRAIN-EFFECT EXPERIMENT



solution heat-treated. Continued coalescence of the precipitate (over-aging), which we assume occurred when the solution heat-treatment was omitted, resulted in decreased effectiveness of the dispersion and lowered strength (56 ksi). Increased strain from rolling a thicker plate apparently caused over-aging to an even greater degree (48 ksi), although the solution heat-treatment still improved the strength (53 vs. 48 ksi).

Rolling at the higher temperature (1400 C) permitted more rapid precipitate growth because of increased diffusion rates than was possible at 1100 C, and the ultimate tensile strength for the solution heat-treated material dropped from 60 to 52 ksi. The increased strain, which was a result of rolling from 620 mils at 1400 C, increased the rate of precipitate growth nullifying the effects of re-solution heat-treatment. The ultimate tensile strength for both sheets was 49 ksi.

4.4.6 Summary

The strengthening of the MTC alloy by strain-induced precipitation of TiC has been qualitatively confirmed by the preceding considerations.

Conclusive metallographic evidence or other quantitative data are desirable but were not obtained. More thorough analysis of the mechanism awaits additional knowledge of the quantity and type of phases present at the temperatures and strain conditions under investigation. The importance of rolling temperature, in-process heat treatment, and amount of deformation have been amply demonstrated.

5.0 PROCESS FOR MTC SHEET

Based on our development work, process specifications were prepared for 40-mil MTC sheet. These specifications, PF-32, PF-33, and PF-34, appear in Appendix III. The processing steps are:

1. Mix required amounts of molybdenum, titanium hydride, and carbon powders.
2. Press into billets.
3. Sinter to at least 92% of theoretical density at 2300 C in dry hydrogen.
4. Breakdown-roll at 1400-1430 C from 1.5 inches to 400 mils.
5. Re-solution heat-treat for two hours at 1700 C (plate recrystallizes).
6. Cross-roll at 1100 C to 150 mils.

7. Straight-roll at 1100 C to 60 mils.
8. Etch to 55 mils.
9. Straight-roll cold to 40 mils.

Etching near the finished size lowers the ductile-brittle transition temperature by an average of 25 C. We etched in a solution, one part by volume 49% HF solution and nine parts by volume concentrated HNO_3.

Breakdown rolling at 1300 C results in split plate at 400 mils. If breakdown rolling is done at or above 1475 C final rolling must be done at a rather high temperature, ~500 C, to avoid failure.

It is important that the lead and lag ends of the sheet are trimmed after cross-rolling. These ends are thinner than the rest of the sheet, and if they are not trimmed the incidence of edge cracking is high upon subsequent straight-rolling.

During the contract period we had an opportunity to demonstrate the feasibility of breaking down sintered MTC by methods other than rolling. Figure 12 shows, in addition to a rolled plate, a plate reduced 50% in height by press-forging in one pressing at 1500 C, and a rod reduced 77% in area by extrusion in a Dynapak at 1625 C.

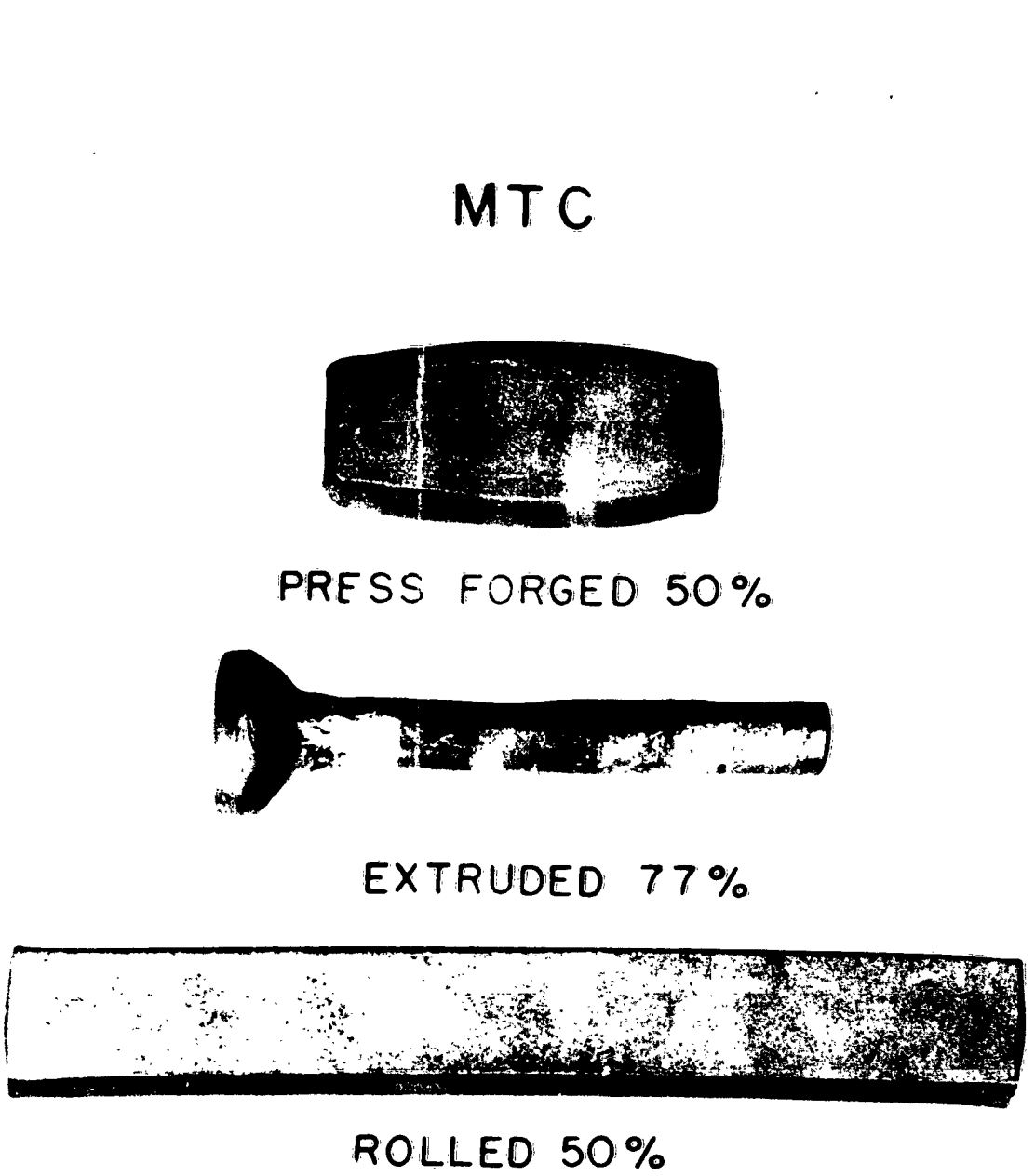


Fig. 12. MTC broken down from sintered billets by press-forging and by rolling at 1500 C and by extruding in a Dynapak at 1625 C.

Eight 40-mil MTC sheets were produced and delivered to the Bureau of Naval Weapons. Six sheets were 6" x 20", and two sheets were about 7-5/8" x 17-1/8". The sheets were rolled from eight sintered billets prepared from the same powder mix. Mixing, pressing, and sintering were done according to specifications PF-32 and PF-33, and the data appear in Appendix IV. Two of the eight billets, Nos. 29-15 and 29-16, were rolled according to specification PF-34. The other six billets, Nos. 29-3, -7, -9, -10, -11, and -13, were rolled first and by a somewhat different shcedule. These billets were broken down at 1475 C instead of at 1400 C, the sheets were etched from 45 to 40 mils instead of from 60 to 55 mils, and the final rolling was done at ~500 C instead of at room temperature. The two sheets rolled according to specification PF-34 were more finely fibrous and had a better surface appearance than those of the other six. The rolling data appear in Appendix IV.

6.0 EVALUATION OF MTC SHEET

We performed no exhaustive statistical evaluation of the properties of MTC sheet. However, we did evaluate some of the sheets which were sent to the Bureau of Naval Weapons and others which were produced in a similar manner. The evaluation consisted of the determination of composition, recrystallization temperature, tensile properties, and

ductile-brittle transition temperature. Some properties were evaluated enough times to give an idea of the degree of reproducibility. Early in the development work some stress-rupture data were obtained on sheet processed with no re-solution heat treatment.

6.1 Composition

Chemical analyses for six of the eight sheets delivered to the Bureau of Naval Weapons appear in Table XI and are summarized below.

Elements	Average	Range
Titanium	0.50%	0.45-0.54%
Carbon	260 ppm	220-310 ppm
Oxygen	33 ppm	15-110 ppm
Nitrogen	12 ppm	2- 22 ppm
Hydrogen	1 ppm	1- 2 ppm
Molybdenum	Bal.	-

Changes in composition during processing are shown below:

Element	Mix	Billet	Sheet
Titanium	0.50%	-	0.50%
Carbon	870 ppm	280 ppm	260 ppm
Oxygen	900 ppm*	80 ppm	33 ppm
Nitrogen	-	10 ppm	12 ppm

* Weight loss of Mo powder in hydrogen.

TABLE XI

COMPOSITION OF FINAL MTC SHEETS

Number	Titanium, %	Ave.	Carbon, ppm	Ave.	Oxygen, ppm(a)	Ave.	Nitrogen, ppm(b)	Ave.	Hydrogen, ppm(c)
29-3	0.48,0.53	0.50	280,260	270	59,50,20(c)	43	22, 8,12(c)	14	2
29-7	0.48,0.52	0.50	250,220	240	31, 18(c)	24	10,11,16(c)	12	2
29-9	0.48,0.53	0.50	290,230	260	110,71,15(c)	65	13,12,16(c)	14	1
29-10	0.47,0.54	0.50	280,250	260	32, 15(c)	24	2,15,15(c)	11	1
29-11	0.45,0.53	0.49	310,260	280	14, 20(c)	17	9, 8,17(c)	11	1
29-13	0.47,0.51	0.49	290,220	260	25, 26(c)	26	10,13,12(c)	12	1
29-15	-	-	-	-	-	-	-	-	-
29-16	-	-	-	-	-	-	-	-	-
Ave.		0.50		260		33		12	1

a Determined by inert-gas-fusion method unless otherwise indicated.
b " " micro-Kjeldahl " " " "
c " " vacuum-fusion.

The amounts of carbon and oxygen lost during processing are in the proportion corresponding to the reaction

$$2C + O_2 \rightarrow 2CO$$

which took place during the sintering cycle[4].

We do not know why the oxygen content of the sheet is apparently lower than that of the sintered billet.

6.2 Recrystallization Temperature

The 50%-recrystallization temperature was determined from hardness measurements on sheet samples annealed for one hour at various temperatures and was subsequently verified by metallographic examination. The hardness values are in Table XII, and they are plotted as a function of annealing temperature in Figure 13. The 50%-recrystallization temperature ranges from 1200-1220 C.

Figure 14 shows photomicrographs of two of the eight as-rolled sheets sent to the Bureau of Naval Weapons. One sheet was rolled according to specification PF-34. In this sheet the fibers are finer than those of the other sheet, which was rolled by the original schedule. Figure 15 shows photomicrographs of sheet annealed for one hour at 1200 C and 1300 C. The structure resulting from annealing at 1200 C is about 50% recrystallized.

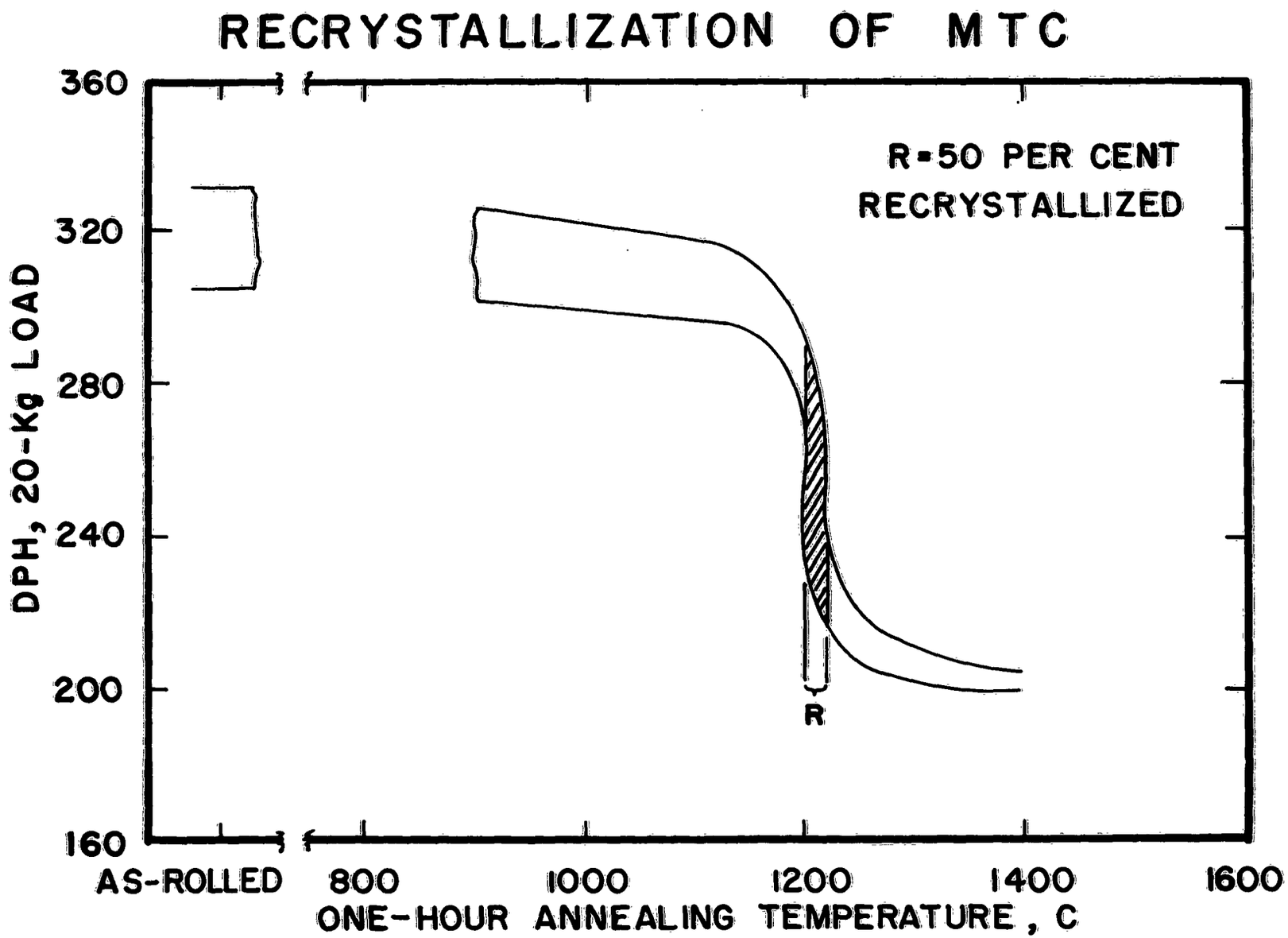
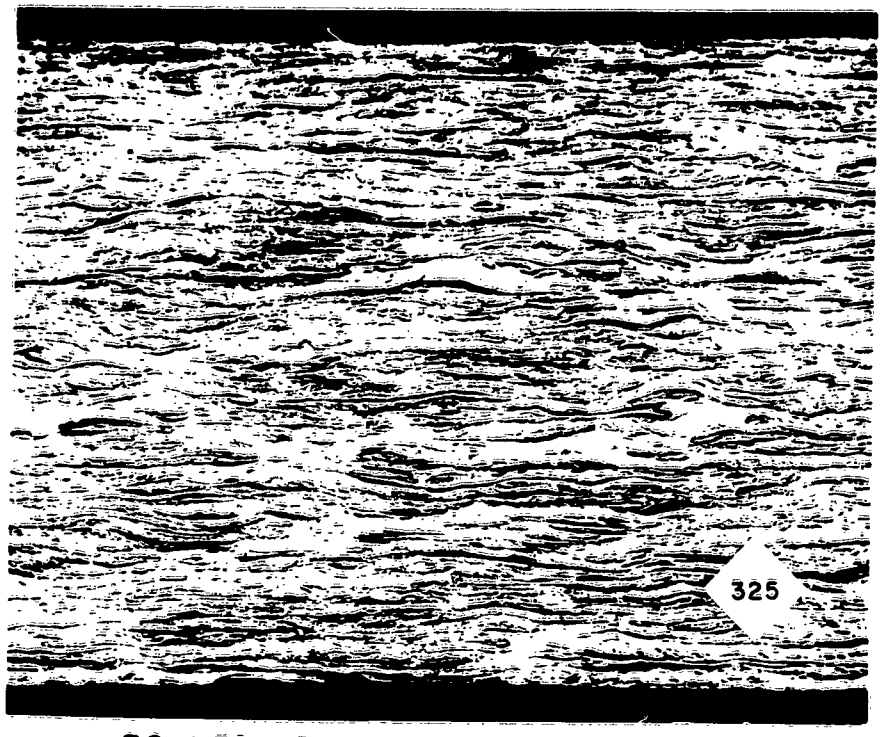
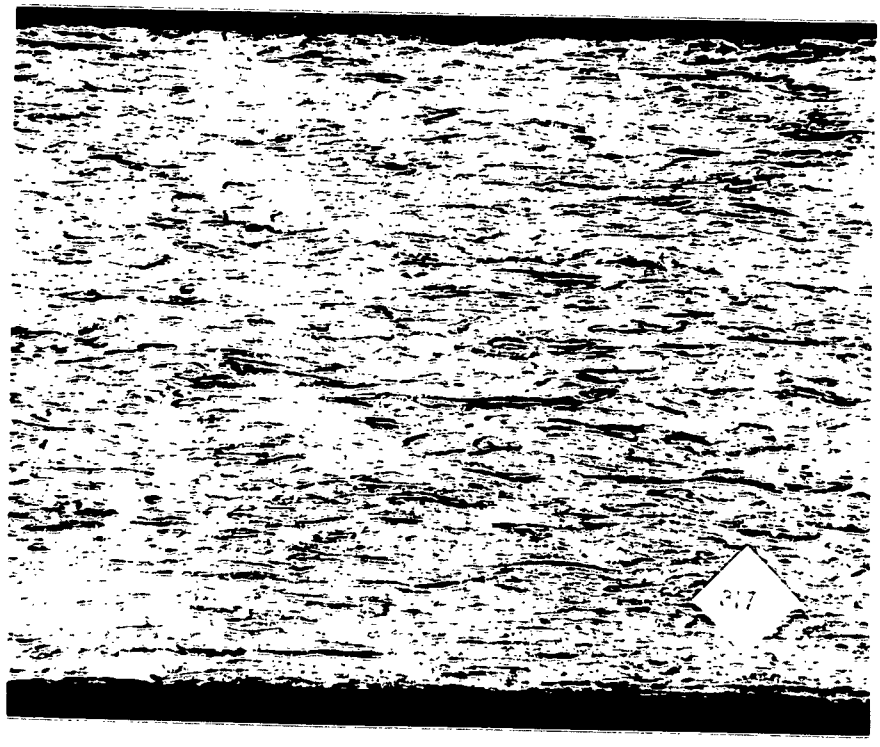


Fig. 13. Recrystallization temperature of 40-mil MTC sheet as determined by hardness measurements.

AS - ROLLED MTC



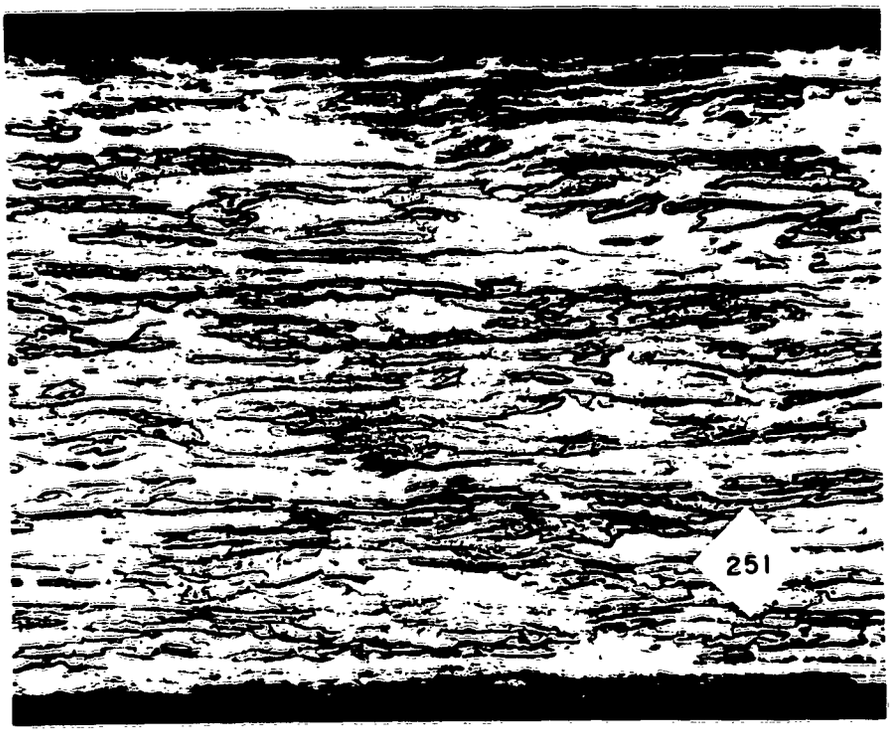
ROLLED BY ORIGINAL SCHEDULE



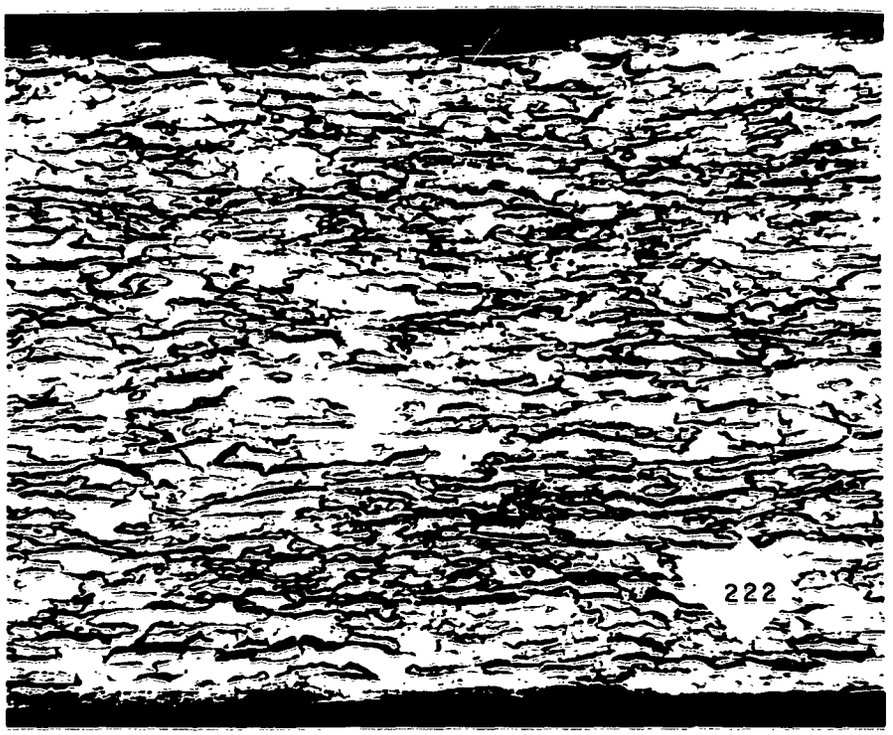
ROLLED BY SPEC. NO. PF-34

Fig. 14. Longitudinal structures of as-rolled 40-mil MTC sheet at 85X. Number on photomicrograph is DPH.

ANNEALED MTC



ONE HOUR AT 1200 C



ONE HOUR AT 1300 C

Fig. 15. Longitudinal structure of annealed 40-mil MTC sheet at 85X. Sheet annealed for one hour at 1200 C is about 50% recrystallized. Number on photomicrograph is DPH.

TABLE XII

HARDNESS OF FINAL MTC SHEET

Sheet No.	DPH_{20}-kg After One Hour at Indicated Temperature, C						
	As-Rolled	1100	1150	1200	1250	1300	1400
29-3	312	303	301	273	217	208	203
29-7	331	294	296	274	221	213	200
29-9	329	312	315	288	218	213	200
29-10	325	317	312	258	207	204	199
29-11	331	300	308	278	222	203	204
29-13	331	308	300	261	215	208	199
29-15	305	-	-	-	-	-	-
29-16	317	-	-	-	-	-	-
Average	323	306	305	272	217	208	201

NOTE: One-hour 50%-recrystallization temperature = 1200-1220 C

The average curve of hardness vs annealing temperature for MTC sheet is compared to those for the two base materials, powder-metallurgical molybdenum and arc-cast Mo-0.5Ti sheets, in Figure 16. MTC and arc-cast Mo-0.5Ti sheets are about 50% recrystallized within the same temperature range. This range is about 180 C above that for powder-metallurgical molybdenum sheet. The hardness of MTC sheet begins to drop sharply at about 1150 C, whereas, that of arc-cast Mo-0.5Ti sheet begins to drop at about 1020 C.

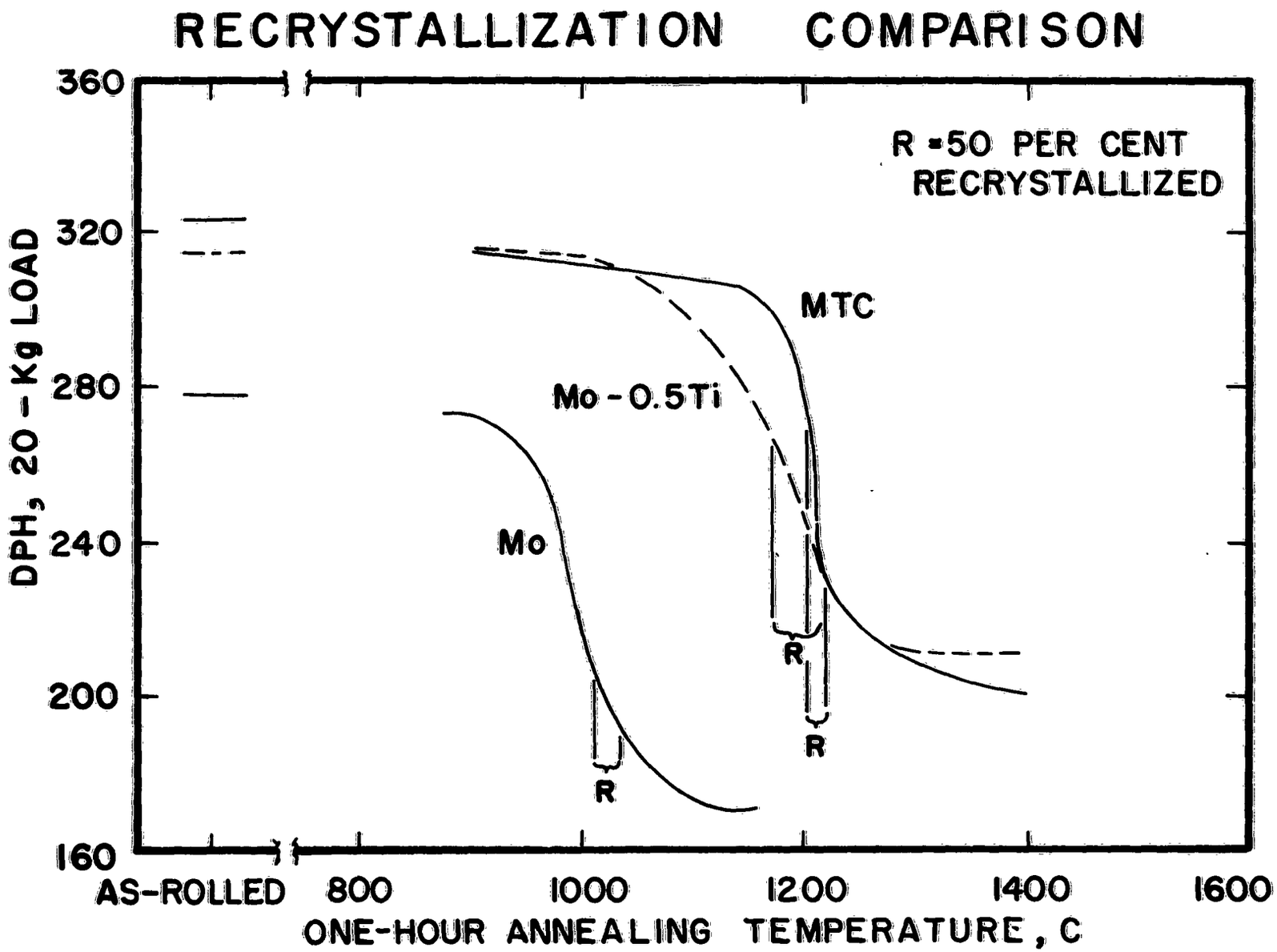


Fig. 16. Comparison of recrystallization temperatures, as determined by hardness measurements, of 40-mil sheets of MTC, powder-metallurgical molybdenum and arc-cast Mo-0.5Ti.

6.3 Tensile Properties

Tensile properties of as-rolled sheets were measured in the longitudinal direction at 25, 1095, and 1200 C, and in the transverse direction at 25 and 1200 C. In addition tensile properties of recrystallized sheets were measured at 25 and 1315 C. The data are in Table XIII. Strengths and elongations in the transverse direction were respectively higher and lower than those in the longitudinal direction.

Tensile properties are plotted as a function of test temperature in Figure 17. The ultimate tensile strengths range from 76 ksi at 1095 C in the as-rolled condition to 19 ksi at 1315 C in the recrystallized condition. The strengths of MTC sheet are compared to those for the two base materials in Figure 18. The targets for high-temperature strength for fabricable molybdenum alloys, established by the Refractory Metal Sheet Rolling Panel, are also included. MTC sheet is considerably stronger than arc-cast Mo-C.5Ti sheet at 1095 and 1200 C, but at 1315 C the strengths are about equal. We expect that at 1095 and 1200 C arc-cast Mo-C.5Ti sheet could be as strong as MTC sheet if a proper re-solution heat treatment was incorporated. At 1095 C, MTC sheet meets the Refractory Metal Sheet Rolling Panel's target strength. However, it falls far short of the target at 1315 C.

TABLE XIII - TENSILE PROPERTIES OF 40-MIL MTC SHEET[a]

Test Temp. C	Sheet No.	As-Rolled Longitudinal UTS ksi	As-Rolled Longitudinal YS,0.2% Offset,ksi	As-Rolled Longitudinal Elongation %	As-Rolled Transverse UTS ksi	As-Rolled Transverse YS,0.2% Offset,ksi	As-Rolled Transverse Elongation %	Recrystallized UTS ksi	Recrystallized YS,0.2% Offset,ksi	Recrystallized Elongation %
25	29-6	151	143	-	163	145	3	-	-	-
		153	145	5	163	147	4			
	29-12	146	137	7	166	151	5	-	-	-
		148	139	5	165	148	4			
	22-10A[b]	157	133	7	165	136	5	-	-	-
	22-11A[c]	154	129	6	157	132	6	-	-	-
	22-12A[d]	157	125	8	156	128	4	-	-	-
	22-6A[f]	-	-	-	-	-	-	81	66	32
	Ave.[g]	153	134	6	161	138	5			
1095	29-6	77	58	8	-	-	-	-	-	-
	29-12	76	58	7	-	-	-	-	-	-
	Ave.	76	58	8						
1200	29-6	61	48	9	-	-	-	-	-	-
	29-12	61	47	8	-	-	-	-	-	-
	22-10A[b]	62	48	10	-	-	-	-	-	-
		53	40	9						
	22-11A[c]	57	45	8	-	-	-	-	-	-
		62	46	9						
	22-12A[d]	61	48	11	-	-	-	-	-	-
		59	44	9						
	29-1	64	51	5	68	56	4	-	-	-
		64	55	5	68	53	4	-	-	-
	Ave.[g]	61	47	8	68	54	4			
1315	29-6	-	-	-	-	-	-	20	17	48
	29-12	-	-	-	-	-	-	18	10	48
	Ave.							19	14	48

a Re-solution treated at 400 mils for two hours at 1700 C unless otherwise indicated.
b Re-solution treated at 400 mils for one hour at 1700 C. Carbon <200 ppm.
c Carbon <200 ppm.
d Re-solution treated at 400 mils for four hours at 1700 C. Carbon <200 ppm.
e At 1450 C for one hour in hydrogen.
f Not re-solution treated.
g Used average of duplicates.

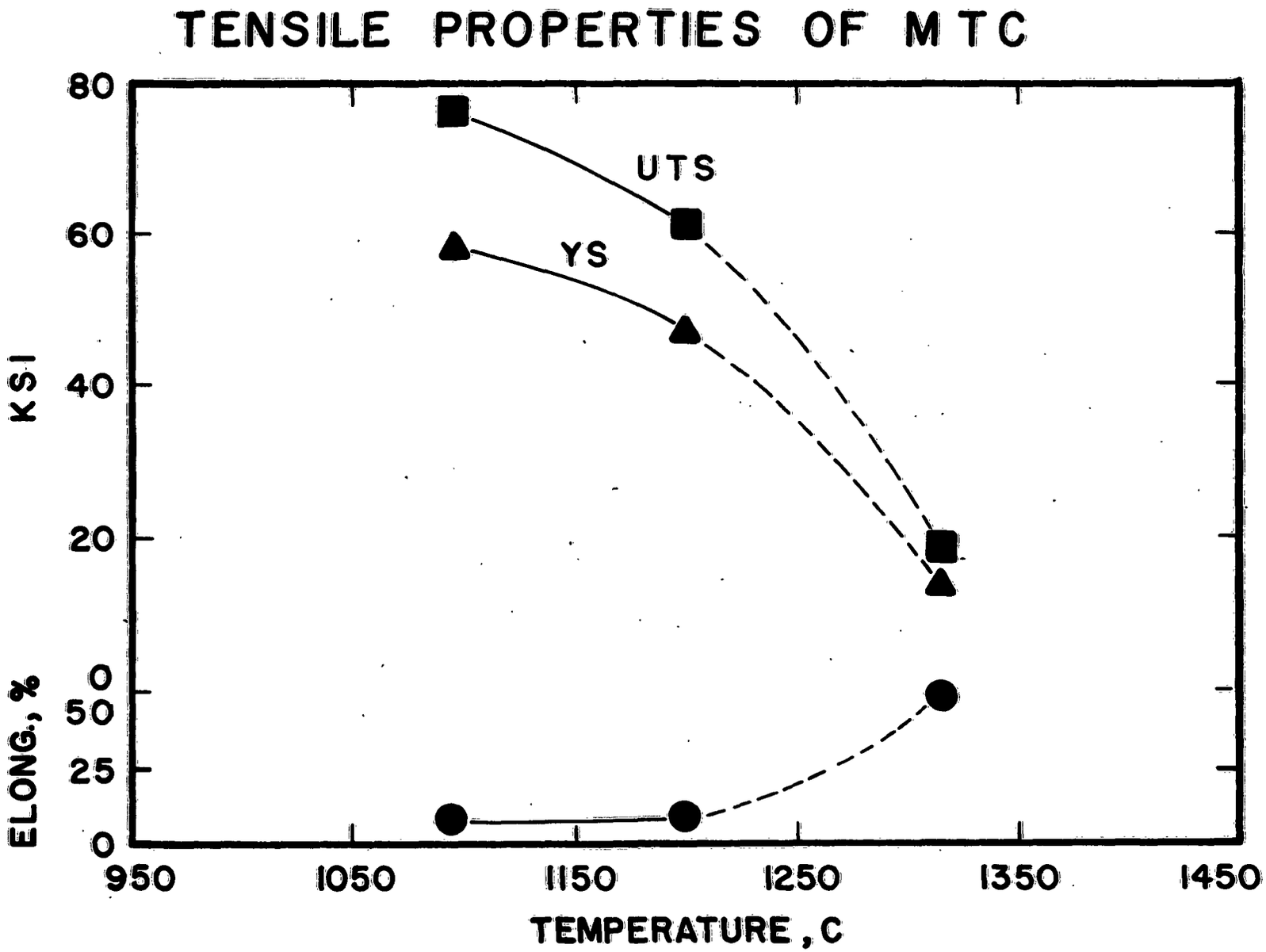


Fig. 17. High-temperature tensile properties of 40-mil MTC sheet. Specimens recrystallized for one hour at 1450 C were used for testing at 1315 C. As-rolled specimens were used for testing at 1095 and 1200 C in the longitudinal direction. Yield strength is 0.2% offset.

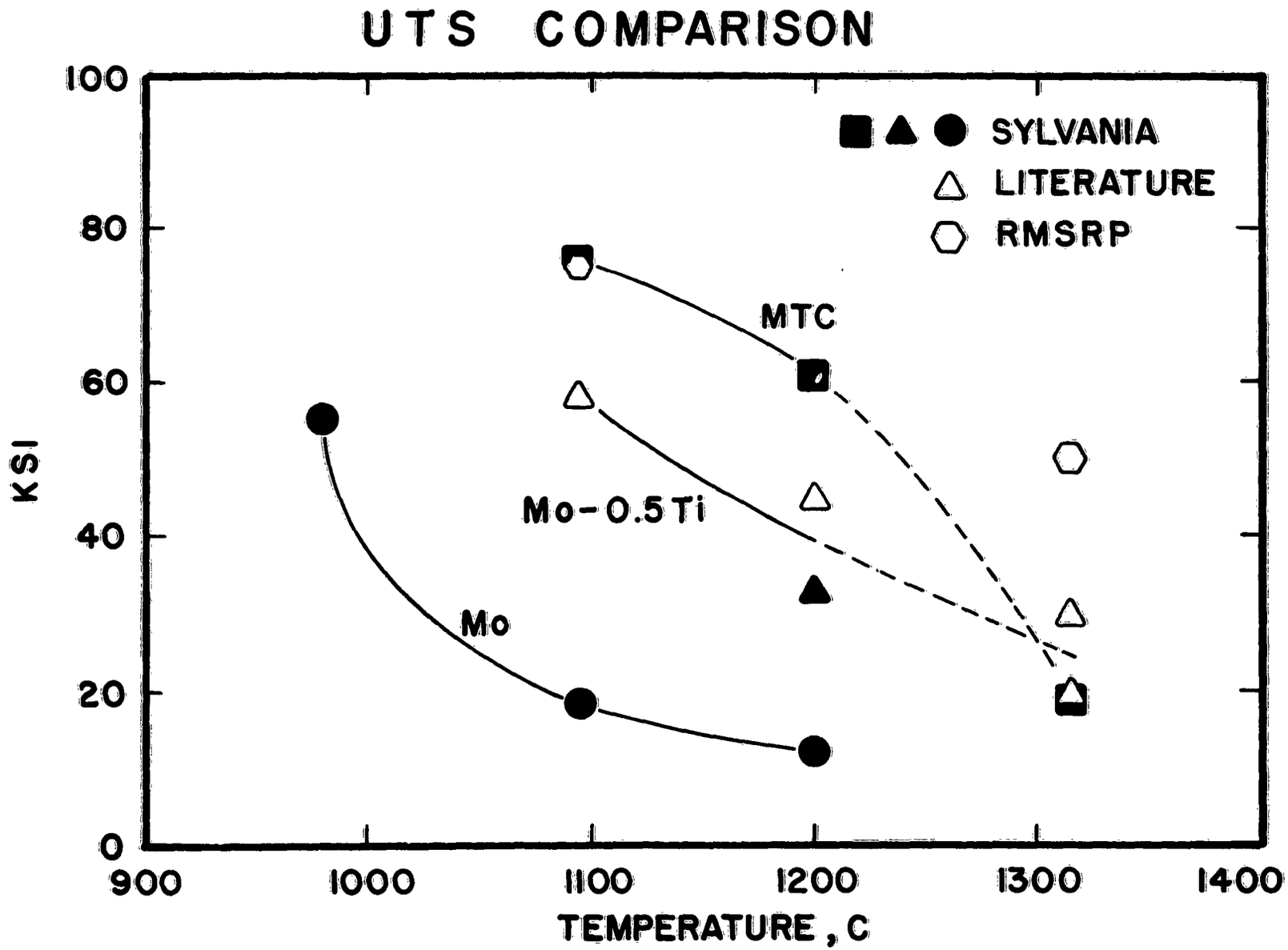


Fig. 18. Comparison of high-temperature ultimate tensile strengths of 40-mil sheets of MTC, powder-metallurgical molybdenum, and arc-cast Mo-0.5Ti to the Refractory Metal Sheet Rolling Panel targets. The Sylvania, RMSRP, and probably the literature values at 1315 C are for recrystallized sheets. Others are for as-rolled or stress-relieved sheets.

6.4 Ductile-Brittle Transition Temperature

The ductile-brittle transition temperature of as-rolled MTC sheet was determined in both the longitudinal and transverse directions by the bend test. The results are in Table XIV and are summarized below.

Direction	Range	Average
Longitudinal	<-75 to -25C	<-40C
Transverse	-25 to 0C	>-17C

Additional development of the rolling schedule is necessary to obtain similar values in both directions.

TABLE XIV

DUCTILE BRITTLE TRANSITION TEMPERATURE OF 40-MIL AS ROLLED MTC SHEET

Sheet No.	Re-solution Time at 1700 C, hr	DBTT, C	
		Longitudinal	Transverse
29-6	2	<-25	0
29-12	2	-25	25
29-15	2	-	>-25
29-16	2	-	-25
22-10A	1	-50	-25
22-11A	2	<-75	0
22-12A	4	-25	0
Ave.		<-40	>-17

6.5 Stress-Rupture

Data on stress-rupture at 1200 C were obtained for MTC sheets and for the base sheets. No re-solution heat treatment was used in processing the MTC sheets, which

were rolled from billets sintered at 2150 C. The data appear in Table XV and are plotted in Figure 19. From this plot the following one- and ten-hour rupture stresses were determined.

Material	Rupture Stress at 1200 C, ksi	
	1 hr	10 hr
Mo	11	-
Mo-0.5Ti	23	15
MTC	40(a)	26

a by extrapolation

MTC sheet ruptures at a higher stress for a given time than does arc-cast Mo-0.5Ti sheet. The rupture stress of MTC sheet decreases at a faster rate than does that of arc-cast Mo-0.5Ti sheet.

6.6 Summary

The properties of powder-metallurgical molybdenum, arc-cast Mo-0.5Ti, and MTC sheet are compared in Table XVI. The Refractory Metal Sheet Rolling Panel's target values are also included. The composition, recrystallization temperature, and ductile-brittle transition temperature of MTC sheet are about the same as those of arc-cast Mo-0.5Ti sheet. At 25, 1095, and 1200 C the ultimate tensile strength and the yield

TABLE XV

DATA FOR STRESS RUPTURE AT 1200 C

Applied Stress, ksi	Rupture Time, Hr — Base Materials: Powder-Met. Mo	Arc-Cast Mo-0.5Ti	MTC[a]
30.0	-	-	2.4
			3.8
			4.4
			5.4
			8.2
			Ave. 4.8
25.0	-	-	6.5
			17.9
			Ave. 9.3
24.2	-	-	16.9
24.0	-	0.43	-
23.0	-	1.70	-
22.5	-	0.94	-
21.5	-	-	27.2
			32.2
			32.2
			Ave. 30.5
18.5	-	3.45	-
17.6	-	5.42	-
17.5	-	-	40.4
17.0	0.28	9.30	-
-			
15.0	-	4.50	-
		20.5	
		Ave. 12.5	
14.0	-	12.8	-
13.0	-	12.5	-
9.60	1.4	-	-
8.00	1.7	-	-
5.15	26	-	-

a No re-solution heat treatment.

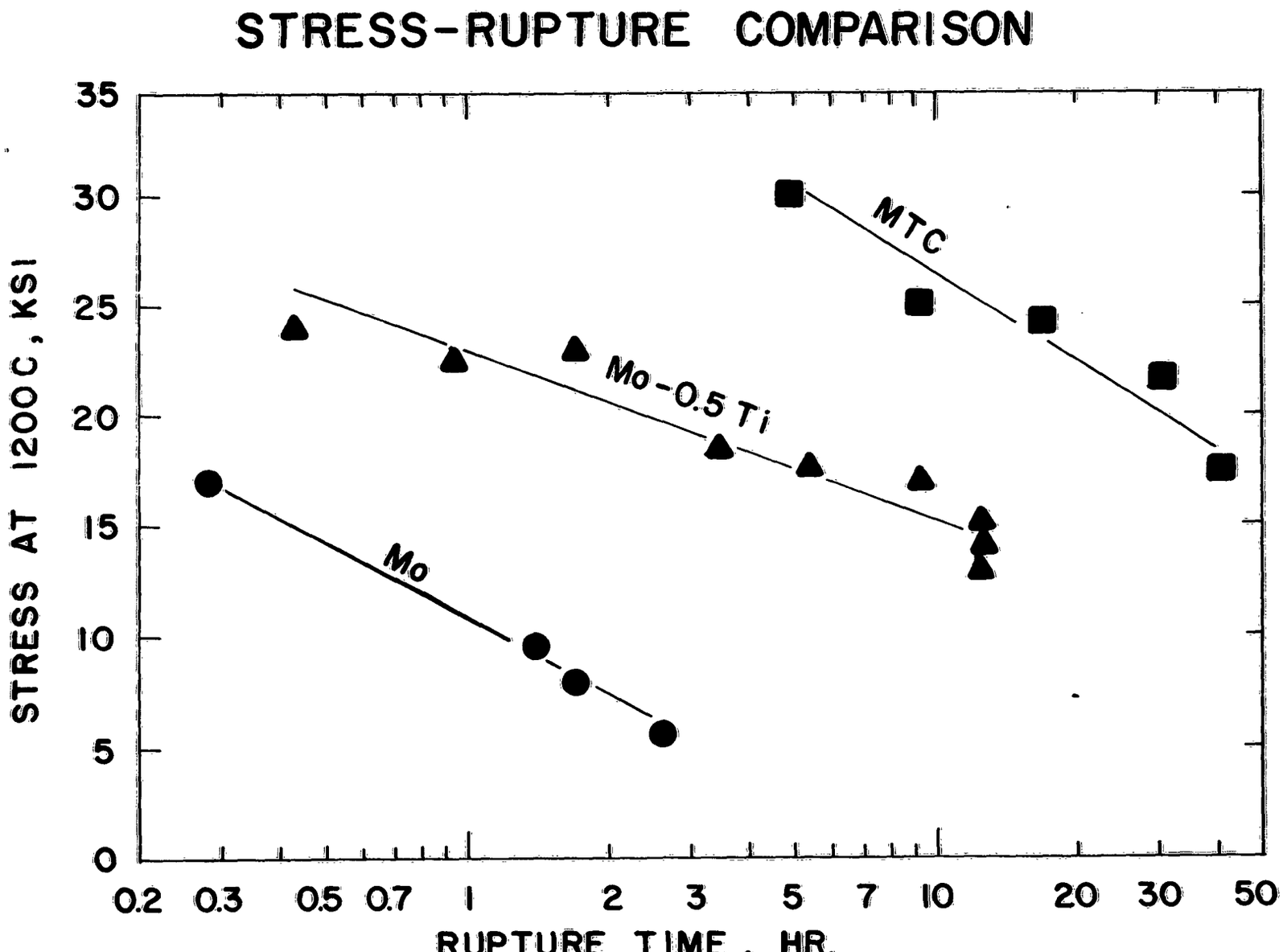


Fig. 1. Stress-rupture comparison of 40-mil sheets of MTC, powder-metallurgical molybdenum and arc-cast Mo-0.5Ti. The MTC was tested in re-solution heat treated.

TABLE VI - PROPERTIES OF 40-MIL Mo AND Mo-ALLOY SHEET

Property	Base Sheet[a] Powder-Met. Mo[b]		Arc-Cast Mo-0.5Ti[c]		MTC[a][b]		RMSRP Target
Composition							
Titanium, %	-		0.49	(4)	0.50	(6)	-
Carbon, %	0.002	(23)	0.028	(4)	0.026	(6)	-
Oxygen, ppm	32	(23)	28	(4)	33	(6)	-
Nitrogen, ppm	28	(23)	14	(4)	12	(6)	-
Hydrogen, ppm	4	(23)	5	(4)	1	(6)	-
Recrystallization Temp. 50% in one hr, C	1020	(11)	1200	(4)	1210	(6)	1425
Ductile-Brittle Transition Temperature, C[d]	<-75	(5)	-42	(4)	<-40	(7)	-40
Tensile Properties[d]							
25 C							
UTS, ksi	127	(15)	128	(4)	153	(5)	-
YS, 0.2% offset, ksi	102	(14)	104	(4)	134	(5)	-
Elongation, %	10	(14)	15	(4)	6	(5)	10
1095 C							
UTS, ksi	18	(1)	58	(e)	76	(2)	75
YS, 0.2% offset, ksi	14	(1)	52	(e)	58	(2)	60
Elongation, %	31	(1)	11	(e)	8	(2)	-
1200 C							
UTS, ksi	12	(2)	33	(4)	61	(6)	-
YS, 0.2% offset, ksi	6	(2)	25	(4)	47	(6)	-
Elongation, %	31	(2)	13	(4)	8	(6)	-
1315 C							
UTS, ksi	-		25	(e)	19[f]	(2)	50[g]
YS, 0.2% offset, ksi	-		10	(e)	14[f]	(2)	35[g]
Elongation, %	-		20	(e)	48[f]	(2)	-

a Numbers in parentheses indicate the number of sheets evaluated to determine values.
b As-rolled condition unless otherwise indicated.
c Stress-relieved.
d Longitudinal properties.
e Literature values, see Table II.
f Recrystallized at 1450 C for one hour.
g Recrystallized condition.

strength of MTC sheet is higher than those of arc-cast Mo-0.5Ti sheet. At 1315 C the strengths are about the same.

The tensile elongations of arc-cast Mo-0.5Ti sheet are higher than those of MTC sheet at 25, 1095, and 1200 C and considerably lower than that of MTC sheet at 1315 C.

MTC sheet meets the Refractory Metal Sheet Rolling Panel's target properties for ductile-brittle transition temperature and ultimate tensile strength at 1095 C. It almost meets the target for yield strength at 1095 C. However, it does not meet the targets for recrystallization temperature, room-temperature tensile elongation, and strength at 1315 C.

7.0 CONCLUSIONS

1. Powder-metallurgical molybdenum-metal oxide sheets, prepared during attempts to translate to sheet our experience gained from developing dispersion-strengthened molybdenum and tungsten wire, showed increases in high-temperature properties over those of molybdenum sheet. However, these increases fell short of those obtained in wire. The highest increases were obtained for Mo-1.0Cr_2O_3, Mo-0.5TiO_2, and Mo-0.5ZrO_2.

2. Powder-metallurgical molybdenum-alloy sheets, whose compositions duplicated those of several commercial and developmental arc-cast alloys, also showed increases in high-temperature properties over those of molybdenum sheet. However, only Mo-0.5Ti-0.03C, ultimately designated MTC, had high-temperature properties which were similar to those of its arc-cast counterpart.

3. The best combination of high- and low-temperature properties was obtained for 40-mil MTC sheet by a processing sequence which included:
   A. adding an amount of carbon to a powder mix of molybdenum and 0.52% titanium hydride such that resultant sheet contained 0.02-0.04%.
   B. sintering green sheet billets at or above 1850 C in dry hydrogen to a density of at least 92% of that of theoretical.
   C. heat-treating rolled plates at or above 1700 C prior to rolling to the final thickness at lower temperatures.
   D. etching the surficial layers from the sheets just before rolling cold to 40 mils.

4. The composition, recrystallization temperature, and the ductile-brittle transition temperature of MTC sheet are similar to those of arc-cast Mo-0.5Ti. MTC's tensile

strength and tensile elongation are respectively higher and lower than those of arc-cast Mo-0.5Ti up to 1200 C.

5. The properties of MTC sheet are marginal when compared to the target properties established by the Refractory Metal Sheet Rolling Panel for a fabricable molybdenum alloy. MTC sheet meets the targets for ductile-brittle transition temperature and ultimate tensile strength at 1095 C. It almost meets the target for yield strength at 1095 C. However, it does not meet the targets for room-temperature tensile elongation, recrystallization temperature, and strength at 1315 C. Additional process development will probably never result in achieving the last two targets.

6. Improvements in strength and recrystallization temperature of MTC sheet relative to those of molybdenum sheet are attributed to the precipitation of TiC induced by strain during rolling.

ACKNOWLEDGMENTS

Among the many personnel who contributed to the work under this contract, the authors wish to acknowledge the following for significant contributions: Drs. J. S. Smith[a], C. D. Dickinson[b], and R. C. Nelson[c], and Messrs. S. Friedman[b] and W. Pollack[b] for technical assistance; Mr. R. H. Rhodes[a] for supervising much of the experimental work; Mr. O. Haines[b] for conducting many of the high-temperature tests; Mr. J. H. Miller[a] for the photographic work.

a Sylvania Electric Products Incorporated, Chemical and Metallurgical Division, Towanda, Pennsylvania.

b General Telephone and Electronics Laboratories Incorporated, Bayside, New York.

c University of Nebraska, Lincoln, Nebraska, formerly with Sylvania-Towanda.

REFERENCES

[1] "Molybdenum Chemicals" Chemical Data Series, Climax Molybdenum Company, New York, September 1, 1954, p. 8.

[2] Bargainnier, R. B., "Development and Production of Improved Molybdenum Sheet by Powder Metallurgy Techniques", Interim Report No. 17, December 31, 1962 (NOas 60-6018-c).

[3] Ibid., Interim Report #13, March 30, 1962.

[4] Ibid., Interim Report #14, August 10, 1962.

[5] Bufferd, A. S., et al., "Hard Particle Strengthening of Refractory Metals for High Temperature Use Through Internal Oxidation", Final Report, New England Laboratories Report to Navy (NOas 59-6223-c) July 1, 1959-June 30, 1960.

[6] "The Metal Molybdenum", edited by J. J. Harwood, ASM, Cleveland, Ohio (1958), "Powder Metallurgy Molybdenum-Base Alloys", R. I. Jaffee, pp 330-364.

[7] Mukherjee, A. K. and Martin, J. W., "Hardening of a Molybdenum Alloy by Nitride Dispersions", Journal of Less-Common Metals, Vol. 2, Oct. 1960, p. 392.

[8] "Molybdenum Sheet Rolling Program", Universal-Cyclops Steel Corp. (for Navy Bureau of Aeronautics) NOas 59-6142-c, Interim Report No. 13, August 25, 1961.

REFERENCES (Cont.)

[9] Semchyshen, M. and Barr, R. Q., "Arc-Cast Molybdenum-Base Alloys", Supplement to Summary Report (NR 039-002) 1957.

[10] Chang, W. H., "A Study of the Influence of Heat Treatment on Microstructure and Properties of Refractory Alloys", Technical Documentary Report No. ASD-TDR-62-211, April, 1962, for Aeronautical Systems Division, Wright-Patterson AFB.

APPENDIX I

TARGET PROPERTIES FOR FABRICABLE MOLYBDENUM-ALLOY SHEET[a]

Requirements	In Optimum Condition	Compl. Recryst.
1. Room-Temp. Tensile		
Ultimate Tensile Strength, ksi	*	*
Yield Strength, 0.2% Offset, ksi	*	*
Elongation, per cent	10	10
2. Elevated-Temp. Tensile		
Temperature, F	2000	2400
Ultimate Tensile Strength, ksi	75	50
Yield Strength, 0.2% Offset, ksi	60	35
Elongation, per cent	*	*
3. Creep-Rupture (State Stress and Elong.) at		
Temperature, F	2000	2400
Rupture-Time, hr	1|10	1|10
4. Recrystallization (in opt. condition) 50% by metallographic observation		
Time	1 hr	
Temperature, F	2600	
5. Notch Sensitivity - ratio[b]	1.0 (RT)	
6. Transition Temperature (In opt. condition)		
in bending 4T	-40	
tensile, notched	State	
smooth	"	
Impact, Charpy	"	
7. Bend ductility (Room Temperature)		
Base metal	1T	
Welded (Weld transverse to bend axis)	4T	

8. STATE FOLLOWING:

Density
Melting Point
Emissivity
Modulus of Elasticity
Thermal Shock Resistance
Creep Properties
Oxidation Resistance & Contamination
Coatability
Experience with 45° Brittleness
Lamination Tendency

* To be furnished

a Report No. 172M, "Report on Refractory Metals Sheet Rolling Panel's Activities", by Materials Advisory Board's Refractory Metal Sheet Rolling Panel, May 22, 1961.

b K_t 6.0, PA 40% (See ASTM Bulletin, January 1960, p 29)

APPENDIX II

TESTING PROCEDURES

CHEMICAL ANALYSIS

1. Sampling - Samples of powder mixes and rolled sheets were taken at random. Samples of sintered billets were cut from a given end, consistent with sintering-furnace orientation.

2. Determinations

   A. Titanium - Determinations were made by a wet chemical method. A 1.4-g sample was dissolved in aqua regia. The solution was evaporated to a low volume and diluted with water. It was made ammonical and $TiO_2 \cdot H_2O$ precipitated. The precipitate was filtered, washed, ignited, and weighed as TiO_2.

   B. Carbon - Determinations were made by a conductometric method in a Leco apparatus. A 0.1-0.4-g sample was used.

   C. Oxygen - Determinations were made by either the vacuum-fusion method with a Fisher's Serfass Gas Analyzer or the inert-gas-fusion method with a Leco conductometric apparatus. A 0.1-0.4-g sample was used.

   D. Nitrogen - Determinations were made by either the vacuum-fusion method or the micro-Kjeldahl method with an all-glass system. A 0.1-0.5-g sample was used.

   E. Hydrogen - Determinations were made by the vacuum-fusion method. A 0.1-0.4-g sample was used.

PHYSICAL TESTING

1. Room-Temperature Tensile. Tests were done on either an Instron or Riehle testing machine. In most cases, tensile test data for a given sheet represent averages of two tests. Either a standard ASTM specimen or a smaller specimen with a 1/4" x 1" gage section was used. The strain rate was 0.005/min to the 0.6% offset. From the 0.6% offset to fracture, a strain rate of 0.05/min was used, except for a few specimens for which a strain rate of 0.02/min was used. In most cases the strain rate was determined by the rate of crosshead separation. Elongation was computed from gage marks on the specimen. Yield strength was determined at the 0.2% offset on a stress-strain curve where the strain was obtained from crosshead movement.

2. High-Temperature Tensile. Specimens with a 1/4" x 1" gage section and pin-type gripping ends were used. The strain rate, as determined by the rate of crosshead separation, was 0.005/min to the 0.6% offset. From the 0.6% offset to fracture, the strain rate was 0.02/min. Tests were done at 980, 1095, 1200 and 1315 C by an Instron testing machine equipped with a Brew vacuum furnace with tantalum heating elements. A vacuum of 10^{-4} mm of mercury or better was obtained for each test. Specimens tested at 1315 C were previously recrystallized by heating at 1450 C for one hour in a hydrogen atmosphere.

Tests at the other temperatures used specimens in the as-rolled condition. Specimens were heated to the testing temperatures in 8-10 minutes and held 5 minutes at temperature before starting the test. The testing temperature, controlled within ± 5 C, was measured by a platinum vs platinum-10% rhodium thermocouple attached to the specimen from a Leeds and Northrup potentiometer. The yield strength was determined at the 0.2% offset on a stress-strain curve with the crosshead movement as a measure of the strain. Elongation was determined by gage marks. Most 1200 C tests were done in duplicate.

3. Stress-Rupture. Stress-rupture tests were done with a 6000-pound capacity Tatnall stress-rupture tester with lever ratio of 4.5:1. Machined sheet specimens were heated in a Kanthol-wound resistance furnace equipped with a vacuum retort. The vacuum ranged from 25 to 50 μ of mercury. The testing temperature of 1200 C was reached in four to five hours and was held for one-half hour before applying the load. Temperature was measured by three platinum vs platinum-10% rhodium thermocouples attached one to each end and one to the center of the gage length. The temperature was controlled within ± 6 C. The specimens had a 1/2 x 2-1/4" reduced section with a 2" gage length. Pin-type grips were used.

4. Recrystallization Temperature. Test coupons, approximately 1/2-inch square, were heated at various temperatures for one hour in hydrogen. They reached temperature in five minutes. The temperature, measured either by a thermocouple located near the boat containing the specimens or by a micro-optical pyrometer sighted on a carbon block in the boat, was controlled within ± 10 C. The diamond pyramid hardness was determined for each specimen from the average of three impressions made by a 20-kg weight. Hardness versus annealing temperature curves were drawn. From these the temperature at which a given sheet was recrystallized 50% in one hour was determined as that temperature corresponding to 2/3 of the hardness drop from the as-rolled to the fully annealed condition. This temperature was then confirmed by metallographic examination.

5. Ductile-Brittle Transition Temperature. This was determined by bending as-rolled sheet specimens, 1" x 3", through an angle of 105° at various temperatures in increments of 25 C until the transition temperature was bracketed. The edges of each specimen were rounded by grinding in a direction parallel to the major axis on a 120-grit wet belt. The bend-test equipment consisted of a mated 75° V-punch and die set operated in conjunction with an Instron testing machine. The punch, with a C.160" (4T) radius, was mounted on the movable crosshead of the testing machine. The punch, moving at

10 in./min, bent the specimens which were located across the top of the die. The die was in the bottom of an insulated chamber which was partially filled with various liquids for testing above and below room temperature. Tests above room temperature were done in either glycerine or mineral oil heated to various temperatures by an immersion heater. The maximum testing temperature was 250 C. Tests down to -75 C were done in isopropanol cooled to various temperatures by dry ice. Testing temperatures were controlled within $\pm$ 3 C. The specimens were held at temperature for three minutes before bending.

The lowest temperature at which specimens could be bent without failure (as observed at 10 X) was considered to be the ductile-brittle transition temperature.

APPENDIX III

PROCESS SPECIFICATIONS FOR MTC SHEET

SPECIFICATION PF-32

PREPARATION OF GREEN MTC SHEET BILLETS FOR CONTRACT NOas 60-6018-c

A. RAW MATERIALS

1. Molybdenum powder:

Molybdenum content, % min. based on metals		99.9
Weight loss in hydrogen at 950 C for one hour, % maximum		0.1
Metallic impurities, ppm maximum,	Al	10
	Ca	10
	Cr	100
	Cu	5
	Mg	10
	Mn	10
	Fe	50
	Ni	100
	Pb	10
	Si	200
	Sn	50
Mesh size		-100
Fisher Subsieve Size, microns		3.5-5.5
Bulk density, g/in^3		20-35

2. Titanium hydride

   Grade E, Metals Hydride Inc., sieved through a 325-mesh screen.
   Fisher Subsieve Size, using density of 3.76 g/cc: 6μ maximum.

3. Carbon

   Grade SP-2, National Carbon Company.

B. PROCEDURE

1. Mixing

   a. Weigh out molybdenum, titanium hydride, and carbon such that

$$\begin{pmatrix}\text{Wt. of } TiH_2\end{pmatrix} = 0.00523 \times \begin{pmatrix}\text{Wt. of Mo}\end{pmatrix}$$
$$\begin{pmatrix}\text{Wt. of C}\end{pmatrix} = 0.00085 \times \begin{pmatrix}\text{Wt. of Mo}\end{pmatrix}$$

b. Mix TiH_2 and C for 0.5 hr by tumbling.

c. Mix the mixture of TiH_2 and C with the Mo for 3 hr in a blender.

d. Store under nitrogen.

2. Pressing

a. Fill a pliable mold of cross section 2-3/8" x 2-3/8" with about 2.8 kg of the mixed powder from 1.d.

b. Press isostatically at 30 ksi and round the corners to a maximum diagonal of 2 inches.

c. Determine the billet densities.

d. Seal billets in polyethylene with a packet of desiccant.

SPECIFICATION PF-33

SINTERING MTC SHEET BILLETS FOR CONTRACT NOas 60-6018-c

A. MATERIALS

Green MTC sheet billets prepared according to Specification PF-32.

B. PROCEDURE

1. Load a single billet into a 2" ID tungsten susceptor in a 50-kw induction furnace.

2. Close the furnace and flush with dry hydrogen (dew point <-35C).

3. Heat billet to 2300 C in about 75 minutes and hold at temperature for 90 minutes.

4. Cool while flushing with dry hydrogen.

5. Submit for rolling all billets with the approximate dimensions 1.5" x 1.5" x 6.5" which satisfy the following requirements:

Density, based on a theoretical density of 10.14 g/cc, %	92 min.
Carbon, ppm	200-400
Oxygen, ppm	$\leq$200
Nitrogen, ppm	$\leq$ 75

The analyses are to be based on samples from a 1/4" slice cut from one end of each billet.

SPECIFICATION PF-34

ROLLING MTC SHEET BILLETS TO 40 MILS FOR CONTRACT NOas 60-6018-c

A. MATERIALS

Sintered MTC sheet billets prepared according to Specification PF-33.

B. PROCEDURE

1. Breakdown rolling at 1400-1430 C.

   a. Straight-roll from ~1.5-inch thick to 590 mils by reductions of about 17%. Five reductions are required. Heat billet for eight minutes prior to the first reduction. Reheat for 3 minutes after each reduction.

   b. Cross-roll to 400 mils by reductions of about 7.5%. Five reductions are required. Reheat for three minutes after all but the last reduction.

   c. Clean in molten caustic.

   d. Trim about 1/2" from each end.

2. Heat-treat for two hours at 1700 C.

3. Intermediate rolling at 1100 C.

   a. Cross-roll to 150 mils by reductions of about 15%. Seven reductions are required. Heat sheet for five minutes prior to the first reduction. Reheat after all but the last reduction for three minutes.

   b. Trim about 1/2" from the lead and lag ends.

   c. Straight-roll to 60 mils by eight reductions. The first six reductions are about 12%; the last two are about 9%. Heat sheet for five minutes prior to the first reduction. Reheat after all but the last reduction for three minutes.

   d. Clean in molten caustic.

4. Etch to 55 mils in warm solution of 1:9 by volume of 49% hydrofluoric acid:concentrated nitric acid.

   (Caution: Solution warms up on contact with sheet and may become violent.)

5. Final rolling at room temperature.

   a. Straight-roll to 40 mils by reductions of $\leq$ 5%.

   b. Trim 1/2-1" from lead and lag ends. Sheet measures 0.040" x 6-7" x >30".

APPENDIX IV

PROCESS DATA FOR MTC SHEET

SYLVANIA ELECTRIC PRODUCTS INC.
CHEMICAL AND METALLURGICAL DIVISION

TOWANDA, PENNSYLVANIA
METALS SECTION

MIXING DATA

Material: MTC Program No.: 01-2820-04 Date: 8-6-62

Specification No.: PF-32 Mix No.: 29 Mix Weight: 70 kg

POWDERS

Powder	Lot No.	Source	Date Produced	Mesh Size	Absolute Density, g/cc	FSSS, μ	Bulk Density, g/in³	Carbon Content, ppm	Wt. Loss in H₂, %	Amount Req'd. in Mix	
										Per Cent	Wt., g or kg
Mo TYPE 390	MOT 1900	SYLVANIA	2/15/62	-100	10.20	5.0	23.4	36	0.09	100	69.6 g
TiH₂ GRADE E	3-3878 A-2	METALS HYDRIDE	—	-325	3.74	4.1	—	1000(MAX)*	—	0.523	364 g
C GRADE SP-1	66-P	AMT'L. CARBON	—	—	—	—	—	—	—	0.085	59.5 g

*ACCORDING TO VENDOR

MIXING

Date Pre-mixed: 8-6-62 Blender Used: 1600cc AMSCO JAR Mixing Time: 0.0 Hr.

Date Mixed: 8-8-62 Blender Used: 4.5 cu. ft. CONICAL @ 22 rpm Mixing Time: 3.0 Hr.

PROPERTIES OF MIX

Absolute Density: 10.04 g/cc FSSS: 5.3 μ Bulk Density: 21.4 g/in³

Analysis: 0.50% Ti, 870 PPM C

Remarks:

cc: Project Engineer

Approved By: R. D. Benjamin

Date: 8-14-62

E-90

SYLVANIA ELECTRIC PRODUCTS INC.
CHEMICAL AND METALLURGICAL DIVISION

TOWANDA, PENNSYLVANIA
METALS SECTION

PRESSING DATA

Material: MTC Program No.: 01-2870-04 Date: 8-15-62

Specification No.: PF-32 Mix No.: 29 Starting Mix Bal.: 70 kg

Mold No.: — Mold Shape: 2¾"x2¾"x13" Pressing Pressure: 30 ksi

Number of Billets/Pressing: 1 Absolute Density of Mix: 10.04 g/cc

Green Density Determined By: MERCURY-IMMERSION Method

Billet No.	Size, in.	TRIMMED Weight, kg	Green Density	
			Measured, g/cc	Per Cent of Theoretical*
29-1	1.8"x1.8"x8.0"	2.53	6.85	68.2
29-2	" " "	2.58	6.86	68.3
29-3	" " "	2.51	6.87	68.4
29-4	" " "	2.44	6.87	68.4
29-5	" " "	2.43	6.86	68.3
29-6	" " "	2.53	6.85	68.2
29-7	" " "	2.60	7.05	70.2
29-8	" " "	2.57	6.84	68.1
29-9	" " "	2.55	6.90	68.7
29-10	" " "	2.54	6.86	68.3
29-11	" " "	2.55	6.90	68.7
29-12	" " "	2.59	6.86	68.3
29-13	" " "	2.56	6.85	68.2
29-14	" " "	2.61	6.95	69.2
29-15	" " "	2.60	6.85	68.2
29-16	" " "	2.61	6.88	68.5
AVERAGE		2.55	6.88	68.5

*Equals, $\frac{\text{Measured Green Density}}{\text{Absolute Density of Mix}}$ X100

Mix Balance After Pressing: 24.8 kg

Remarks: GREEN BILLETS HAVE 2" DIAGONALS

cc: Project Engineer
Sintering Department

Approved By: R. B. Bargainnier

Date: 8-17-62

E-91

SYLVANIA ELECTRIC PRODUCTS INC.
CHEMICAL AND METALLURGICAL DIVISION

TOWANDA, PENNSYLVANIA
METALS SECTION

SINTERING DATA

Material: MTC Program No.: 01-2370-04 Date: 8-20-62

Specification No.: PF-33 Billet Shape: SHEET BILLET Nominal Green Size: 1.8 x 1.8 x 8.0 in.

Sintering Fce.: 50 KW Sintering Temp.: 2300 C Time-at-Temp.: 1.0 HR.

Time-To-Temp.: 1.25 HR. Or Stoke Rate: -- Atmosphere: DRY VIRGIN H_2

Atmo. Flow Rate: 50 cfh Susceptor Size: 2"D x 8" Susceptor Material: TUNGSTEN

Theoretical Density: 10.14 g/cc Sintered Den. Determined By: Hg-IMMERSION Method

Billet No.	Green		Dew Point, C	SINTERED	Sintered Size, in.
	Weight, kg	Density, Per Cent of Theoretical		Weight, kg	
				AFTER SAMPLING	AFTER SAMPLING
29-1	2.63	68.2	-49	2.30	1.5 x 1.5 x 6.5
29-2	2.68	68.3	-45	2.41	" " "
29-3	2.61	68.4	-45	2.34	" " "
29-4	2.44	68.4	—	2.30	" " "
29-5	2.43	68.3	—	2.33	" " "
29-6	2.53	68.2	-40	2.35	" " "
29-7	2.60	70.2	-46	2.42	" " "
29-8	2.57	68.1	-40	2.43	" " "
29-9	2.53	68.7	-42	2.40	" " "
29-10	2.64	68.3	-42	2.40	" " "
29-11	2.55	68.7	-40	2.44	" " "
29-12	2.59	68.3	-40	2.44	" " "
29-13	2.56	68.2	-40	2.40	" " "
29-14	2.61	69.2	-40	2.43	" " "
29-15	2.60	68.2	—	2.51	" " "
29-16	2.61	68.5	—	2.51	" " "
AVERAGE	2.55	68.6	-43	2.41	

SYLVANIA ELECTRIC PRODUCTS INC.
CHEMICAL AND METALLURGICAL DIVISION

TOWANDA, PENNSYLVANIA
METALS SECTION

SINTERING DATA (Cont.)

Billet No.	Sintered Density: Measured, g/cc	Sintered Density: Per Cent of* Theoretical	Grain Count, mm^{-2}	Chemical Analysis: Carbon, ppm	Chemical Analysis: Oxygen, ppm	Chemical Analysis: Nitrogen, ppm	Other
29-1	9.44	93.1	810	270	50	26	
29-2	9.53	94.0	580	290	110	28	
29-3	9.40	92.7	550	310	86	< 1	
29-4	9.52	93.9	—	150 *	120	37	
29-5	9.54	94.1	—	210	210 *	27	
29-6	9.49	93.6	440	270	170	< 1	
29-7	9.49	93.6	440	280	100	8	
29-8	9.37	92.4	740	260	110	7	
29-9	9.44	93.1	580	270	84	20	
29-10	9.52	93.9	460	290	67	9	
29-11	9.52	93.9	430	260	47	10	
29-12	9.55	94.2	450	250	62	9	
29-13	9.53	94.0	500	280	93	13	
29-14	9.53	94.0	390	250	89	8	
29-15	9.52	93.9	—	170 *	180	19	
29-16	9.50	93.7	—	200	220 *	36	
AVERAGE	9.49	93.6	530	250	110	16	

*Equals, $\frac{\text{Measured Sintered Density}}{\text{Theoretical Density}} \times 100$

Remarks: * OUT OF SPEC. - Wanted

cc: Project Engineer

Approved By: R.B. Baganniu

Date: 9-2-62

D-92B

SYLVANIA ELECTRIC PRODUCTS INC.
CHEMICAL AND METALLURGICAL DIVISION

TOWANDA, PENNSYLVANIA
METALS SECTION

ROLLING DATA

Material: MTC Program No.: 01-2370-04 Date: 9-10-62

Specification No.: — Billet Shape: SHEET BILLET Nominal Sintered Size: 1.5x1.5 x6.5 in.

Breakdown Temp. 1426° C Intermediate Temp.: 1100C Final Temp.: ~500 C

Heat Treatments: 2HR. @1700C @0.40" Other Treatments: CHEM-MILL FROM 0.045" TO 0.040"

Billet and Sheet No.	Billet			Sheet		Efficiency,* %
	Weight, kg	Size, in.	Density, Per Cent of Theoretical	Size, in.	Weight, kg	
29-3	2.34	1.5x1.5x6.5	92.7	~0.040x8x29	1.47	62.8
29-7	2.42	" " "	93.6	" " "	1.55	64.1
29-9	2.40	" " "	93.1	" " "	1.58	65.8
29-10	2.40	" " "	93.9	" " "	1.54	64.2
29-11	2.44	" " "	93.9	" " "	1.61	66.0
29-13	2.40	" " "	94.0	" " "	1.52	63.3
AVERAGE	2.40		93.5		1.54 —	64.4

*Equals, $\frac{\text{Sheet Wt.}}{\text{Billet Wt.}}$ X100

Remarks: BREAKDOWN - 18% RA/PASS TO ~0.56" (STRAIGHT ROLL). TRIMMED TO 0.040"X6"X20"
12% RA/PASS TO 0.40" (CROSS ROLL).
INTERMEDIATE - 15% RA/PASS TO ~0.13" (CROSS ROLL).
9-13% RA/PASS TO 0.060" (STRAIGHT ROLL).
FINAL - < 10% RA/PASS TO 0.045" (STRAIGHT ROLL).

: Project Engineer

Approved By: R. B. Baganmin

Date: 9-20-62

E-93

SYLVANIA ELECTRIC PRODUCTS INC.
CHEMICAL AND METALLURGICAL DIVISION

TONAWANDA, PENNSYLVANIA
METALS SECTION

ROLLING DATA

Material: MTC Program No.: 01-2370-04 Date: 9-26-62

Specification No.: PF-34 Billet Shape: SHEET BILLET Nominal Sintered Size: .625 x 6.5 in.

Breakdown Temp. SEE SPEC. C Intermediate Temp.: SEE SPEC. C Final Temp.: SEE SPEC. C

Heat Treatments: SEE SPEC. Other Treatments: SEE SPEC.

Billet and Sheet No.	Billet			Sheet		Efficiency,*
	Weight, kg	Size, in.	Density, Per Cent of Theoretical	Size, in.	Weight, kg	
29-15	2.51	1.5 x 1.5 x 6.5	93.9	0.041 x 7⅞ x 25	1.39	55.4
29-16	2.51	" " "	93.7	0.042 x 7⅞ x 31	1.67	66.5

*Equals, $\frac{\text{Sheet Wt.}}{\text{Billet Wt.}}$ X100

Remarks:

Project Engineer

Approved By: R. B. Bargainnier

Date: 10-4-62

E-93

Contract NOas 60-6018-c Distribution List

Naval Air Material Center
Philadelphia 12, Pennsylvania
Attn: Aeronautical Materials Laboratory

Office of Naval Research
Navy Department
Washington 25, D. C.
Attn: Code 423

Naval Research Laboratory
Washington 25, D. C.

Watertown Arsenal Laboratory
Watertown, Massachusetts
Attn: Mr. S. V. Arnold

Aeronautical Systems Division
Air Force Systems Command
Wright-Patterson Air Force Base
Ohio
Attn: WWRCMP-2

Directorate of Resources
Manufacturing Methods Division
Wright-Patterson Air Force Base
Dayton, Ohio
Attn: LMBM

National Aeronautics and Space Administration
1512 H Street, N.W.
Washington 25, D. C.
(5 copies)

A.E.C., Research Division
Germantown, Maryland

Mr. G. Mervin Ault
Assistant Chief
Materials and Structures Division
Lewis Research Center
National Aeronautics and Space Administration
21000 Brookpark Road
Cleveland 35, Ohio

Dr. Edward Czarnecki, Manager
Materials Mechanics and Structure Branch
Systems Management Office
Boeing Airplane Company
Seattle 24, Washington

Mr. Louis P. Jahnke, Manager
Metallurgical Engineering
Applied Research Operations Propulsion Laboratory
Aircraft Gas Turbine Department
General Electric Company
Evandale, Ohio

Dr. Robert I. Jaffee, Tech. Mgr.
Department of Metallurgy
Battelle Memorial Institute
505 King Avenue
Columbus 1, Ohio

Hughes Tool Company
Materials and Fabrication
Engineering Department
Aircraft Division
Culver City, California

Mr. L. M. Raring, Chief
Metallurgical and Chemical Laboratories
Pratt and Whitney Aircraft
Connecticut Aircraft Nuclear Engine Laboratory
P. O. Box 611
Middletown, Connecticut

Mr. Arnold Rustay
Vice President
Wyman-Gordon Company
North Grafton, Massachusetts

Mr. George A. Timmons
Director of Research
Climax Molybdenum Company of Michigan
14410 Woodrow Wilson Boulevard
Detroit 3, Michigan

Contract NOas 60-6018-c Distribution List

Dr. L. L. Seigle, Manager
Metallurgical Laboratory
General Telephone and Electronics Laboratories Inc.
Bayside 60, New York

Dr. William Rostoker
Assistant Manager
Metals Research Department
Armour Research Foundation
Technology Center
Chicago 16, Illinois

United Aircraft Corporation
Pratt and Whitney Division
East Hartford, Connecticut

Clevite Research Center
540 East 105th Street
Cleveland, Ohio
Attn: Mr. A. Schwope

Thompson Ramo Wooldridge, Inc.
23555 Euclid Avenue
Cleveland 17, Ohio
Attn: Mr. G. N. Guarnieri
Staff R and D

National Research Corporation
70 Memorial Drive
Cambridge 42, Massachusetts
Attn: Mr. J. H. Gardner

Battelle Memorial Institute
505 King Avenue
Columbus 1, Ohio
Attn: Defense Metals Information Center

Aerospace Industries Association
7660 Beverly Boulevard
Los Angeles 36, California
Attn: Mr. H. D. Moran
(3 copies)

Southern Research Institute
917 South 20th Street
Birmingham, Alabama

Massachusetts Institute of Technology
Cambridge 39, Massachusetts
Attn: Professor N. J. Grant
Metallurgy

Union Carbide Metals Company
Technology Department
P. O. Box 580
Niagara Falls, New York
Attn: Dr. R. W. Fountain

Universal-Cyclops Steel Corp.
Bridgeville, Pennsylvania
Attn: Mr. P. C. Rossin

E. I. DuPont de Nemours and Co.
Pigments Department
Experimental Station
Wilmington 98, Delaware
Attn: Dr. E. M. Mahla

Fansteel Metallurgical Corp.
2200 Sheridan Road
North Chicago, Illinois
Attn: Dr. A. B. Michael

Wah Chang Corporation
Box 366
Albany, Oregon
Attn: Mr. E. Baroch

Westinghouse Electric Corp.
Metals Plant
P. O. Box 128
Blairsville, Pennsylvania
Attn: Mr. F. L. Orrell

Westinghouse Electric Corp.
Lamp Division
Bloomfield, New Jersey
Attn: Dr. R. H. Atkinson

Contract NOas 60-6018-c Distribution List

Firth Sterling Inc.
3113 Forbes Street
Pittsburgh 30, Pennsylvania
Attn: Dr. C. H. Toensing

Lockheed Missile and Space Division
3251 Hanover Street
Palo Alto, California
Attn: Dr. T. E. Tietz and
Mr. Roger Perkins

Republic Aviation Corporation
Farmingdale, L. I., New York
Attn: Mr. E. A. Simkovich
Advanced Systems and Research

University of California
Los Alamos Scientific Laboratory
P. O. Box 1663
Los Alamos, New Mexico
Attn: Dr. R. D. Baker

Raytheon Company
Missile Systems Division
Andover, Massachusetts
Attn: Mr. W. R. Johnson
Metallurgist

Marquardt Aircraft Company
16555 Saticoy Street
P. O. Box 2013 - South Annex
Van Nuys, California

Mr. E. J. Baker
Inspector Naval Material
Post Office Building, Room 17
Kingston, Pennsylvania

Mr. J. Maltz
RRMA 22
Department of the Navy
Bureau of Naval Weapons
Washington 25, D. C.
(3 copies)

Department of the Navy
Bureau of Naval Weapons
RMMP-23
Washington 25, D. C.

Department of the Navy
Bureau of Naval Weapons
DIS-3
Washington 25, D. C.
(3 copies)

Department of the Navy
Bureau of Naval Weapons
SP-271
Washington 25, D. C.

General Electric Company
Research Laboratory
P. O. Box 1088
Schenectady, New York
Attn: Dr. D. W. Lillie

Dr. Joseph R. Lane
Staff Metallurgist
Materials Advisory Board
National Academy of Sciences
National Research Council
2101 Constitution Avenue
Washington 25, D. C.

Mr. A. V. Levy and
Mr. Robert E. Matt
Materials Research and Development
Solid Rocket Plant - Dept. 5810
P. O. Box 1947
Sacramento, California

Mr. B. S. Payne, Manager
New Products Research
The Pfaudler Company
Rochester 3, New York

Stauffer Metals Company
1202 S. 47th Street
Richmond, California
Attn: Mr. Jack K. Y. Hum

Contract NOas 60-6018-c Distribution List

Thiokol Chemical Corporation
Brigham City, Utah
Attn: Mr. J. A. Bryson

Union Carbide Nuclear Corporation
Building 9202
P. O. Box "Y"
Oak Ridge, Tennessee
Attn: Mr. A. C. Neeley

Mr. J. T. Stacy
Senior Group Engineer
Boeing Airplane Company
Aero-Space Division
P. O. Box 3707
Seattle 24, Washington

Thermionic Products Company, Inc.
Interhaven Avenue
Plainfield, New Jersey
Attn: Mr. V. O. Allen

Mr. Robert F. Zuehlke
Ladish Company
Cudahy, Wisconsin

Dr. Russell C. Nelson
Room 202J, Richards Hall
Mechanical Engineering Department
University of Nebraska
Lincoln 8, Nebraska

Allegheny Ludlum Steel Corp.
Brackenridge, Pennsylvania
Attn: Mr. P. A. Santoli
Res. and Dev. Labs.

Army Research Office
Box CM, Duke Station
Durham, North Carolina

Bureau of Ships
Navy Department
Washington 25, D. C.
Attn: Codes 342B and 1500

Curtiss-Wright Corporation
Wright Aero. Division
Woodridge, New Jersey
Attn: Mr. J. Sohn

Denver Research Institute
University Park
Denver 10, Colorado
Attn: Dr. W. E. Mueller

General Motors Corporation
Allison Division
Indianapolis, Indiana
Attn: Mr. D. K. Hanink